Master's thesis

NTNU
Norwegian University of Science and Technology
Faculty of Humanities
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Fanny Sikström

A Whale As Old As Time

Interpreting radiocarbon dates of whale bones

Master's thesis in Archaeology
Supervisor: James H. Barrett
Co-supervisor: Bente Philippsen & Marie-Josée Nadeau
May 2023

Slettbakur from Jón Guðmundsson and his natural history of Iceland

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Abstract

This thesis aims to demonstrate the relevance of radiocarbon dating whale bones discovered in archaeological contexts and the significance of a species-specific ΔR. It also aims to test the theory that whale bones' protein and fat content would provide different ages. Its goal is, therefore, to radiocarbon date the collagen and lipid of 20 bone samples collected from the three species North Atlantic Right Whale (*Eubalaena glacialis*), Bowhead Whale (*Balaena mysticetus*), and North Atlantic Gray Whale (*Eschrichtius robustus).*

When radiocarbon dating the bones of marine mammals, you must take into account the marine reservoir effect. The marine reservoir effect varies temporally and spatially, and there is also variation within marine species. So, local corrections expressed as ΔR are necessary in order to get correct calendar dates. By using a Bowhead Whale from a known age context, it was possible to establish a species-specific ΔR value expressed as the interval (-225, -20) or as (-124 ± 59). With this ΔR correction, it was possible to correct the dates of all the Bowhead samples.

Another thing to consider when radiocarbon dating is the turnover rate of the material that gets dated. This will be especially important to consider when dating long-lived species, such as some species of whale like the Bowhead Whale. The protein, or collagen, will have a significant inherent age due to the old age of the whale, whilst the fat, or lipid, is likely to have a fast turnover rate and reflect the time the whale died. Comparing the date of these two fractions has the possibility to provide insight into how the lifespan of a species affects its dating. While the lipid extraction was successful with 7 samples, they all resulted in significantly older dates than should have been possible. FTIR-spectroscopy showed that this is most likely due to finely-grained sediments being dated and not the actual lipid. Therefore, it was not possible to compare the fractions. The result of this project was 19 successfully dated collagen samples. Six of these were Bowheads and got ΔR corrected and placed in a historical context. The difference in dating results before and after ΔR correcting shows the significance of a species-specific ΔR.

Abstrakt

Dette prosjektet har som hensikt å demonstrere relevansen av å datere hvalbein fra arkeologisk kontekst og viktigheten av å benytte en artsspesifikk ΔR. Samt teste teorien at proteinet og fettet fra hvalbein vil gi ulike dateringer. Målet med oppgaven vil derfor være å radiokarbon datere kollagenet og lipidene tatt fra 20 beinprøver hentet fra de tre hvalartene Nordkaper (*Eubalaena glacialis*), Grønlandshval (*Balaena mysticetus*), og Gråhval (*Eschrichtius robustus).*

Når bein fra sjøpattedyr blir datert må det tas hensyn til marin reservoar effekten. Marin reservoareffekten varierer i både tid og rom, og det finnes også variasjon innad ulike arter. Dermed er det nødvendig å benytte lokale kalibreringsverdier kalt ΔR for å få korrekte dateringer. Ved å benytte en Grønlandshval prøve tatt fra kontekst hvor bruksperioden er kjent var det mulig å etablere en arts spesifikk ΔR verdi. Denne verdien er uttrykt som intervallet (-225, -20) eller som (-124 ± 59). Denne gjorde det mulig å korrigere Grønlandshval dateringene.

En annen faktor å vurdere er forfalsshastigheten til materialet som blir datert. Dette er spesielt relevant under datering av langlevende arter, slik som Grønlandshvalen. Proteinet, eller kollagenet, vil ha en betydelig innebygd alder som et resultat av hvalens alder. Mens fettet, eller lipidene, har en hurtigere forfalsshastighet og reflekterer tiden hvalen døde. Å sammenligne dateringen av disse har potensialet til å gi ny innsikt inn i hvordan alderen til arten påvirker dateringen. Lipid ekstraksjonen var vellykket på 7 av prøvene, men dateringene var langt eldre enn hva som skulle vært mulig. FTIR-spektroskopi viste at dette var grunnet finkornet sediment i prøven ble datert i stedet for lipidene. Derfor var det ikke mulig å sammenligne dateringene. Resultatet av dette prosjektet ble 19 daterte kollagenprøver. Seks av disse tilhørte Grønlandshvalen som ble ΔR korrigert og plassert i historisk kontekst. Forskjellen i resultatene før og etter ΔR-korrigering viser viktigheten av å benytte artsspesifikk ΔR.

Acknowledgements

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Chapter 1: Introduction

This master thesis aims to demonstrate the relevance of radiocarbon dating whale bones discovered in archaeological contexts and the significance of a species-specific ΔR.

It also aims to test the theory that whale bones' protein and fat content would provide different ages. The protein, or collagen, will have a significant inherent age due to the old age of the whale, whilst the fat, or lipid, is likely to have a fast turnover rate and reflect the time the whale died. The inbuilt age of the animal is rarely considered during dating, which is why doing so might provide new insight into the radiocarbon dating of marine mammals.

One goal will therefore be to radiocarbon date and compare the collagen and lipid of 20 archaeological whale bone samples from the North Atlantic. The whale taxa chosen are the three species considered to have been important targets during medieval and early modern whaling: North Atlantic Right Whale (*Eubalena glacialis*), Bowhead Whale (*Balaena mysticetus*), and Gray Whale (*Eschrichtius robustus*). The Bowhead Whales (*Balaena mysticetus*) are extremely long-lived, with a possible lifespan of two centuries (Kovacs et al., 2020), presumably creating a problem like the «old wood effect», which amplifies the marine reservoir effect when radiocarbon dating. The North Atlantic Right Whale is also a very long-lived species, with the oldest known individual being around 70 years (Philip K. Hamilton et al., 1998; Kraus & Rolland, 2007). What makes them noteworthy to include is that they were probably the primary target of whalers in medieval and modern times. Therefore, there will likely be many whale bones in the archaeological record from this species. The Gray Whale, on the other hand, has a shorter life span and is included mainly to compare with the longer-lived whales. Whales are potentially appropriate for a lipid-based study as their bones contain enormous amounts of fat when fresh, the bone of some species containing more than 50% lipid, and some of this might survive in archaeological specimens (Charpentier et al., 2022).

A second goal of this project is to establish a species-specific ΔR for the Bowhead Whale (Balaena mysticetus). While much research has been conducted to develop species-specific ΔRs for marine animals in the North Atlantic and polar regions, as of now, none have succeeded in doing so for the Bowhead by using samples of known ages. This is one of the best methods for establishing ΔR values (Ascough et al., 2005).

Whale bones are underrepresented in the archaeological record and are often deemed unimportant, and overlooked. Primarily because they were difficult to interpret without advancements in methods such as ZooMS and aDNA. By providing dates for whale bones from archaeological contexts, I will help broaden the understanding of when whales of the relevant species were hunted and thus contextualise whaling as a culturally situated practice.

1.1 Materials and Method

The whale bones used are from several archaeological collections across the North Atlantic acquired by Youri van den Hurk. Most of the samples originate from the Netherlands; the rest are from Spain, Norway, Sweden, and Svalbard (Norway). The samples went through collagen mass peptide fingerprinting (ZooMS) before dating, to determine the taxon.

With the exception of one sample, none of the material is from a known age context. As a result, only a ΔR for this Bowhead Whale sample will be estimated and used to estimate the ΔR of the other Bowhead Whale individuals. The other species are included to compare the lipid and collagen dates. Some of the material is classified as Balaenidae rather than Bowhead Whale or North Atlantic Right Whale since the bones could not be identified down to a species level through ZooMS (Buckley et al, 2014).

Collagen and lipid will be extracted from the whale bones. The collagen extraction will follow already established techniques and protocols. However, a new extraction protocol for the lipid will bedeveloped in conjunction with this project. Both the lipid and collagen will be dated at the National Laboratory for Age Determination in Trondheim.

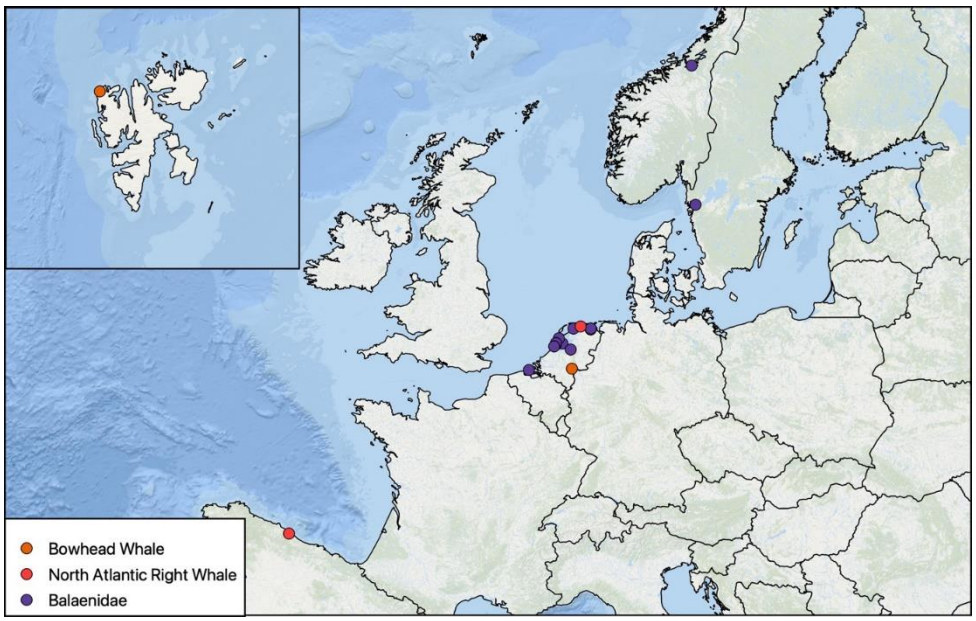

Figure 1. Map showing where the samples were collected from. The Gray Whale samples were found in the North Sea and have no known collection site. Map by Author.

1.2 Key terms

1.2.1 Radiocarbon dating

Radiocarbon dating has been one of the most impactful scientific methods introduced to archaeology. When introduced, it was immediately embraced by the archaeological community and became standard practice soon thereafter (Liden & Eriksson, 2013). It is one of the most reliable and commonly used techniques for dating organic material (Taylor & Bar-Yosef, 2014) with fairly high precision (Liden & Eriksson, 2013).

Briefly explained, Radiocarbon dating measures the ratio of the remaining ^{14}C, or radiocarbon, left in organic material and uses it to determine how long since the material stopped taking in carbon. Carbon has three naturally occurring isotopes, two stable ^{12}C and ^{13}C, and one unstable, which is the one used in radiocarbon dating ^{14}C, (Taylor & Bar-Yosef, 2014) Radiocarbon is continually formed by a series of nuclear reactions that takes place in the upper atmosphere. The ^{14}C isotope gets taken up by organisms during their lifetime and makes radiocarbon dating possible (Ramsey, 2008). The materials that can be dated by radiocarbon are organic materials, which means that they have been part of the biosphere and therefore taken up carbon (Ramsey, 2008). The most commonly dated archaeological materials include bone and charcoal. Carbon enters the biosphere via photosynthesis in green plants, including algae, and is subsequently introduced to animals either directly or indirectly through the food chain (Ramsey, 2008). When an organism dies, it ceases to exchange carbon with the biosphere and no longer takes up the ^{14}C isotope (Ramsey, 2008). Since the ^{14}C isotope is unstable, the concentration of ^{14}C in the animal slowly decreases due to radioactive decay (Taylor, 2001; Ramsey, 2008). The measurement of the remaining ^{14}C content creates the basis for radiocarbon dating (Taylor, 2001).

The measurement of ^{14}C content is done by Accelerator Mass Spectrometry (AMS), which has the sensitivity to detecting specific elements, like ^{14}C, according to their atomic weight (Ramsey, 2008).

During their life, some animals lay down carbon polymers, and some of them cease exchanging carbon with the rest of the organism after they have been formed and retain a detailed temporal isotopic record (Ramsey, 2008). In contrast, some have a faster turnover rate and will contain a relatively recent isotopic record (like fat). In structures where there are also carbon-containing mineral components (i. e. Bone)

the turnover rate is low. This means the isotopic record might not be contemporary with the time of death (Ramsey, 2008).

1.2.2 Calibrating radiocarbon dates

The radiocarbon date or ^{14}C age, expressed as BP[1], are not actual dates but measures of ^{14}C content in the sample. The ^{14}C age is accompanied by an error estimate expressed as plus or minus (±) standard deviation (Banning, 2000; Taylor, 2001). There is not a direct correlation between ^{14}C age and calendar years. This is due to the ^{14}C levels have not been constant over time and varied greatly. Therefore, radiocarbon years need to be calibrated into calendar years in order to get an accurate age estimation. This is done by using computer programs able to compare the ^{14}C age against the calibration curve (Banning, 2000). The calibration curve is determined by measuring ^{14}C concentrations of known age samples (Heaton, et al., 2020).

However, it is not possible to calibrate all samples with the same curve. The concentration of ^{14}C in the ocean and atmosphere differ from each other, creating an offset in the ^{14}C age in organisms from marine and terrestrial environments.

1.2.3 Marine reservoir effect and establishing a ΔR

The offset in the ^{14}C age between organisms that derive their carbon from the terrestrial environment and organisms that derive their carbon from the marine environment is known as the Marine reservoir effect (MRE). When dating samples which obtained their ^{14}C in the marine environment, they will need a marine-specific calibration curve. Instead of using the atmospheric-based IntCal20 curve, the Marine20 curve is used when calibrating radiocarbon ages for marine samples. Marine20 is a marine radiocarbon age calibration curve that represents the non-polar global-average marine record of radiocarbon from 0-55 000 cal BP (Heaton et al., 2020). It should be noted that this curve is not always suitable for calibration in polar regions and might not provide an accurate calibration for the samples from, for example, Svalbard. This is because the marine reservoir ages and their potential changes over time are

[1] BP stands for Before Present.
The present is calculated as 1950, which is when American chemist W.F developed the method. (Libby, 1965)

anticipated to be more significant because of the substantially higher variability in ocean circulation and air-sea gas exchange caused chiefly by variations in sea ice extent and wind strength (Heaton et al., 2020).

This marine offset is not constant and varies greatly spatially, so to accurately calibrate marine samples, the global marine calibration curve alone will not be enough. Local deviations from it, expressed as ΔR, are therefore vital in order to get an accurate dating. Species-specific ΔR values may also sometimes be necessary, as marine reservoir effects can differ between species within a given body of water. Diet, feeding depth, and migratory behaviour are all contributing factors that affect the ^{14}C date of a marine organism—often resulting in significant variation (Dury et al., 2022).

There are three main approaches used to establish ΔR: known age samples, tephra isochrones, and paired terrestrial/marine samples from sealed archaeological contexts. In this study, the method that will be used is using known age samples. These are samples where the calendar death of the organism is well documented, allowing for the comparison of contemporaneous atmospheric and marine radiocarbon ages (Ascough et al., 2005). The advantage of this method is that the date has the potential to be highly accurate and precise. But it is heavily dependent on appropriate samples that are previously well-documented. Most appropriate samples would then likely be from more modern collections. This means analysing prehistoric samples might be close to impossible with this method.

There are already several studies done in attempts to establish reservoir corrections for marine mammals. Dyke et al. (2011) tried to establish a reservoir correction for the Bowhead Whale *(Balaena mysticetus)* by dating ear bones and comparing the ages with driftwood from comparable elevations, terrestrial plant detritus from a stratigraphic section. Then compared, the ages of the bones collected from the youngest (lowest) raised beaches with the ages «Expected», given the elevation of the shoreline (Dyke et al., 2011). The study suggested that a ΔR correction of about -200 years is appropriate for normalised age determinations on bone collagen from the Bowhead Whale in the region studied (Dyke et al., 2011). The normally applied correction in the region studied is based on marine molluscs and is around -400 years. This shows firstly how important it is to determine a species-specific ΔR in order to get the correct dates. Furthermore, it also shows that the carbon in the collagen in Bowhead Whales *(Balaena mysticetus)* derives from their diet instead of the marine bicarbonate, which is the carbon source

for the mollusc shells (Dyke et al., 2011). More recently, Pienkowski et al. (2022) tried to establish revised regional ΔR values for the molluscs and cetaceans from the Barents Sea. It was done by using previously established ^{14}C dates on pre-bomb live-collected materials available from the Barents Sea and adjacent regions. The result was ΔR = -158 ± 43 ^{14}C years for baleen whales with the Marine20 curve (Pieńkowski et al., 2022). Baleen whales are highly diverse with different migration patterns and foraging strategies which would cause variations in the required adjustment. So this correction might not be suitable for some species of baleen whales.

So as of now, no species-specific marine reservoir correction for radiocarbon age determinations on Bowhead Whales by direct dating of specimens harvested from known locations prior to 1954 CE has been established until this study.

1.3 Significance and Contribution

Getting the whale bones dated and putting them into a correct historical context will contribute to our understanding of which species were hunted, where they were hunted, and when they were hunted. This can provide new insight into the human exploitation of these important resources. To accurately date marine samples establishing species-specific ΔR values is essential. The whales' migratory nature makes using local ΔR values futile. The only way to date these animals accurately is by using a species-specific ΔR and quantifying the uncertainty or possible range of this value. Therefore, the ΔR will be helpful for future dating of Bowhead Whale samples and to accurately date the samples used in this project.

Testing the dating of lipids from the whale bone will help with quality-controlling dating results from these species. It may also give new insight into how radiocarbon dates are affected by the biology and longevity of these animals. If the dates prove to be different, we might also (within the limits of radiocarbon precision) be able to gain further insight into the age of death of the individual. This can raise new questions such as; Did the whalers deliberately target older specimens? If they did, can we see a change in the ages of the whales as a response to hunting?

1.4 Roadmap

This thesis is divided into seven chapters. The second chapter is centred around whaling in history to provide context for the time periods the whale bone dates will fall under, which will aid in interpreting the dates. As well show that the whale has been relevant even if there is a lack of archaeological material. The third chapter goes into the selected whale species in more depth as knowledge of the species and where they live, eat, etc., are relevant when dating. The fourth chapter is divided into two parts. The first is about the selection of material and why the specific samples were chosen for this project. The second part goes through all the different methods used and how the lipid method was developed. This includes extraction and pre-treatment of the collagen and lipid, using OxCal to establish a ΔR, and FTIR-Spectroscopy. The fifth chapter will go present the results. First, for the Collagen, then the ΔR corrected Bowhead dates, and then the lipids. The interpretation of the Bowhead dates will be discussed in Chapter 6. The final chapter will summarise the findings and draw conclusions.

Chapter 2: Background

In order to give background information on the historical context the whale bones will fall under, this chapter will give a brief overview of the history of whaling in the North Atlantic. It will also go through the reason for whaling not having a large presence in the archaeological material. This thesis is focused on the North Atlantic Right Whale (*Eubalena glacialis*), Bowhead Whale (*Balaena Mysticetus*), and Gray Whale (*Eschrichtius robustus*), so the whaling activities discussed will mainly concern the ones that were focused on these species. As most of the material originates from the Netherlands, I choose to put a greater focus on Dutch Whaling activities to provide context for the chosen samples.

2.1 Human Exploitation of Whales

Cetaceans have been used as food and resource for millennia. Whale exploitation began independently among coastal communities around the world, and by the 15th century, it had grown into a massive international enterprise (Rey-Iglesia, et al., 2018; Hennius., 2022). From which we still can feel the effects of this exploitation today, with several of the species being close to or completely extinct. In the North Atlantic and Arctic, several of the species are either extinct or critically endangered as a result of the whaling activities that took place. This sub-chapter aims to give a brief overview of the whaling activities relevant to this region and thesis. Therefore as much of the material originates from the Netherlands, there will be a larger focus on the Dutch whaling activities.

2.1.1 Opportunistically vs. deliberate Hunting

In most studies done on the exploitation of cetaceans, the distinction is often made between opportunistic utilisation and deliberate hunting. Opportunistic killing can refer to several practices. Often it is used to refer to the use of stranded animals. But also to the killing of individuals who would find themselves near the coast occasionally or that would accidentally trap themselves in fishing nets (Rodrigues., et al., 2016). What categorises all of these as opportunistic hunting is that these captures would not be planned and rather be the response to vulnerable prey available. While those whales were killed for the resources they could provide, it was not with the immediate intention of economic earning. Deliberate hunting is most often used synonymously with the word whaling. Whaling is often a definition reserved for deliberate, active

hunting for whales, often with specific species targeted. Deliberate meaning it was an operation with the intent to kill and exploit the whale, often with specific techniques and gear -as well as a planned progress for processing, storing, and transporting of the meat and blubber obtained. Whaling is, therefore, an operation requiring a higher level of social organisation and preparation than opportunistic killings (Rodrigues., et al., 2016).

Until recently, it has previously been difficult to distinguish between the two in the archaeological record until recently. With the ability to determine the species from the bones found (through methods like ZooMS), it is now possible to make an assumption if the material stems from either opportunistic or deliberate hunting/whaling. When the material consists of several specific species, and also when these species are coastal, it is fair to assume that the material is a result of deliberate hunting rather than the use of stranded individuals.

2.1.2 Prehistoric Exploitation of Whales

Figure 2. Depiction of whale in the rock art of Alta. The whale is interpreted as a North Atlantic Right Whale (Eubalaena glacialis), as it is the only species in the surrounding oceans blowing in a split spout. (Gjerde, 2017) The panel is dated between 1000BCE-200CE. (Tansem, 2010)

It is often assumed that prehistoric exploitation of whales was mostly limited to the opportunistic use of beached animals (Szabo, 2008; Van Den Hurk et al., 2022). The exploitation of a beached animal would not need dedicated technology or skill; therefore, it is not expected to find supporting material for this in the archaeological record (Rodrigues., et al., 2016). The lack of bones in the material is likely due to difficulties with transporting large bones to settlements (Seersholm et al., 2016; see 2.2). Therefore, it is difficult to determine to which degree the exploitation of whales took place in prehistory. We are aware, though, that the event has occurred, as indicated by various sites in Europe, North America, and Africa (Seersholm et al., 2016).

The first people considered to have begun actively taking advantage of whales in the North Atlantic are the Inuit; indigenous people who lived in the Arctic regions of Alaska, Canada, and Greenland. They have long been among the world's most heavily maritime-oriented hunter-gatherers. Beginning some five millennia ago, these occupants of

predominantly ice tundra coasts adjusted to the relative impoverishment of the terrestrial ecosystem (Whitridge, 1999). They did this by acquiring the technologies and modes of socioeconomic organisation for hunting a wide variety of marine mammals in open water and from the sea ice (Whitridge., 1999). This includes the development of toggling harpoons that appear about 4000 BCE (Seersholm et al., 2016).

In Scandinavia, recent research has been undertaken that indicates that whaling might have taken place earlier in the North Atlantic than previously thought. During the Iron Age in Scandinavia, there was an increase in whale bones being used as raw material, especially for the making of gaming pieces. In Swedish burials from the Late Iron Age, the number of gaming pieces made from whale bones is numerous and dominated from 550CE until the end of the Viking Age (1000AD). This coincided with an increase in slab-lined pits used for blubber processing and the use of whale bone for other types of artefacts (Hennius, Gustavsson, Ljungkvist, & Spindler, 2018). The technology suitable to take down a whale was not present in Norway pre-500 CE, but in the Iron Age Iron, iron spearpoints and arrowheads that would potentially be suitable are more frequent in weapon burials between 650- and 950 CE (Hennius., et al., 2022). As they would have the technology available, as well as the necessary social organisation it is completely plausible that deliberate whaling for economic purposes took place in Scandinavia in the late Iron Age.

This might have been the same for other parts of Europe during the same period (European Middle Ages). Commercial whaling would have been possible due to the existence of established trading networks, long-distance seafaring, and advanced technology and skills (Rodrigues et al., 2016).

2.1.3 The beginning of commercial whaling

The beginning of commercial whaling was pioneered by the Basques in the Cantabrian Sea in the 11th Century (Rey-Iglesia et al., 2018). However, literary evidence of the earliest commercial hunting is from 670 CE when a French basque supplier signs a contract for 40 barrels of whale oil to an Abby in Jumiege, in France (Hennius., et al., 2022). It is still debated whether they were the first commercial whalers or not; but they did pioneer large-scale whaling in Europe.

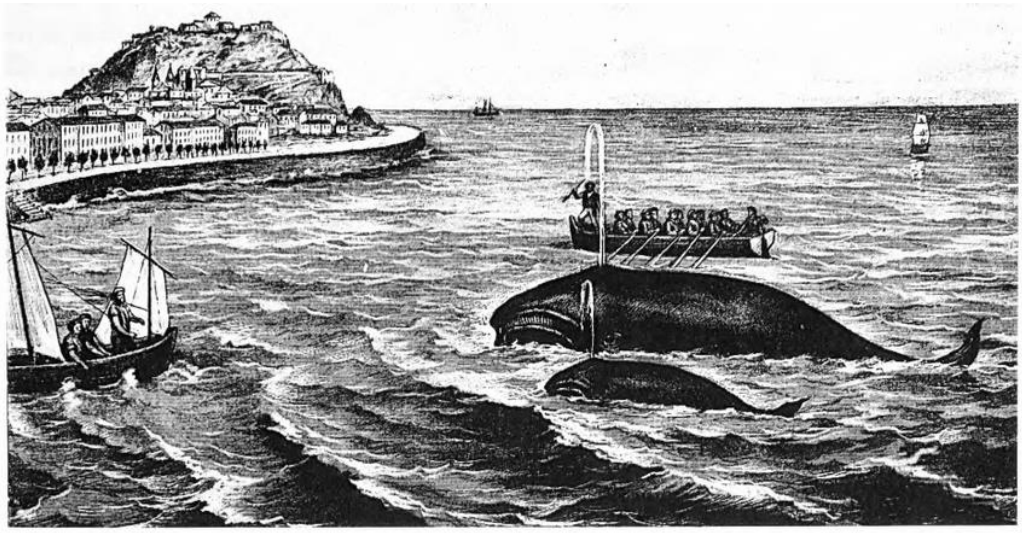

Figure 3. Old whaling in the Bay of Biscay (Aguilar, 1986).

The Basques practised shore-based whaling. They would wait until the whales were close to the shore, at which point watchmen in watchtowers would alert the whalers with smoke signals or drum pounding (Aguilar, 1986; Rey-Iglesia et al., 2018; van den Hurk, 2020). The whalers would then man their small, open, clinker-built boats, known as «chalupas». The chalupas were 6-8 m long with a crew consisting of 5-10 whalers (Rey-Iglesia et al., 2018). They approached the whale, and when they were within firing distance of the whale, the harpooner threw his weapon at the whale. The harpoon was thrown hard in order to penetrate as deep as possible, not with the intention of killing the animal but to keep it from escaping. The line of the harpoon was attached to a drogue, which could be a buoyant block of wood or the boat itself (Rey-Iglesia et al., 2018). When the whale got tired of dragging the drogue, the whalers would approach again either to fasten a new drogue or to deliver the killing blow. The buoyancy of the whale made it easy for the whalers to tow the carcass back to shore. The Basques made it a point to target the calves. They were easier taken, and they knew the mother would come to its aid, making her an easy target as well (Aguilar, 1986).

Their main target species was the North Atlantic Right Whale (*Eubalena glacialis*), which approached the coast of Spain during its winter migration. The migration corresponded with their calving season in November-April (Aguilar, 1986). Other species were also targeted, such as the Bowhead Whale (*Balaena mysticetus*), Gray Whale (*Eschrictius*

robustus), and to a lesser extent, most likely due to them being offshore species, the Humpback Whale (*Megaptera Novaeangliae*) and Sperm Whale (*Physeter Macrocephalus*) -the sperm oil being an especially valuable resource worth acquiring, in spite of the greater effort needed to catch this species.

The shore-living species, such as the North Atlantic Right Whale and Gray Whales, made an easy catch as they favour close in-shore waters, especially during calving (Rey-Iglesia et al., 2018). The North Atlantic Right Whales' size and weight makes it slow-moving and easier to approach as well.

After hauling the whales to shore, they were processed as quickly as possible to avoid the whales' rapid decay process. The Basques would utilise all the parts of the whale that could be extracted. The blubber was melted into oil, the meat salted and consumed, and the bones were used to make tools, utensils, and for construction (van den Hurk, 2020; Rey-Iglesia et al., 2018).

Figure 4. Basque Whale-Fishing. (Whale Fishing Fac Simile of a Woodcut in the Cosmographie Universelle of Thevet in Folio Paris 1574, 1574)

The effects of the Basques' whaling began to show in the mid-14th century. When the local whale populations began to decline, the Basques were forced to extend their hunting grounds. At first, they began whaling off the Irish coast between 1353 and -1561 (van den Hurk, 2020). In 1412 they established a whaling station off the coast of western Iceland and began hunting the Bowhead Whale (van den Hurk, 2020). Around 1530 they continued their hunt for the Bowhead Whale and began exploiting populations in the Strait of Belle Isle, Canada (van den Hurk, 2020). Whaling in North America declined at the end of the 17th century for several reasons (Kruse, 2020), but an important reason is that the other European actors also established whaling activities in the Arctic.

2.1.4 Arctic whaling

Europe's population grew dramatically towards the start of the 17th century. As a result, the price of whale goods increased (Joost C. A. Schokkenbroek et al., 2008). Natural fat from plants was still accessible, but the prices had risen, making the production of plant oil scarce. It created a demand for a substitute, shifting the focus to oils obtained from marine mammals (Joost C. A. Schokkenbroek et al., 2008). The Basques whaling had declined, leaving a shortage of whale products in the European market (Kruse, 2020). At first, the Europeans travelled to Russia and the Norwegian coast to buy whale oil. During the time when Barentz discovered Bjørnøya and Spitsbergen at the end of the 16th century, there was a high demand for whale products. This led to an opportunity for Basque harpooners to find work and for several traders to expand their reach towards the north. Europeans followed in the Basques' footsteps and moved their whaling operations towards the Arctic. By the 1620s, several nations had established themselves in the Arctic whaling scene, and the English and Dutch had even set up stations along the coast of Svalbard and Jan Mayen (Degroot, 2022).

2.1.5 Dutch Whaling in the Arctic

The Dutch ventures into the Arctic began with the Dutch mariner Willem Barentsz's discovery of Svalbard in 1596 (Prestvold, 2001). In the years following, abundant wildlife, including a plethora of whales in the seas surrounding the islands, was reported (Prestvold, 2001). The Dutch headed for the Arctic region around 1612 (Joost C. A. Schokkenbroek et

al., 2008). They had hired numerous Basque Whalers with them who were well-versed in the techniques and procedures required to capture the whales (Prestvold, 2001).

The Dutch experienced heavy competition and rivalry from the other European countries. They would mainly compete with the English over whaling rights and hunting grounds. This would repeatedly result in confrontations and even warfare among the competitors. This resulted in the hunting regions being partitioned, and the Dutch could operate in the north of Spitsbergen (Joost C. A. Schokkenbroek et al., 2008).

The Dutch excelled during this first period in bay whaling. Their target was the Bowhead Whale (*Balaena mysticetus*) which kept to the bays of Spitsbergen. The Bowhead Whale are giant whales with thick coats of blubber on their bodies, making them a slow-moving target (see 2.1.3). In likeness with the North Atlantic Right Whale (*Eubalaena glacialis*) would also stay afloat when killed, which was an advantage when attaching the carcass to the boats when dragging it back to shore. The whale was killed by using Basque techniques (see 2.1.3) and brought back to shore for processing. The Dutch established oil cookeries along the coast of northern Spitsbergen. The largest and most well-known is Smeerenburg.

Smeerenburg, «blubber town», was the largest whaling station and served as the headquarters for Dutch whaling, while the hunt took place along the coast of Svalbard in the first half of the 17th century. Smeerenburg was located near the whale hunting grounds in the fjord, had a suitable port with appropriate anchorage, and provided escape routes for ships, should ice move into the fjord (Prestvold, 2001). It was in use from 1619. In the beginning, Smeerenburg was not much more than a few tents and loose boilers but it transformed into a more permanent settlement when houses got built in 1619 (Hacquebord, 1985). By the 1630s, it had grown into a small town. At its height, it is estimated that it housed somewhere between 100- and 200 people (Hacquebord, 1985). The town housed 7 or 8 oil cookeries that all were used at the same time when the production was at its highest (Hacquebord, 1985).

The whales were towed to Smeerenburg for processing. While still in the water, the flensers removed strips of blubber with flensing knives (Hacquebord, 1985). These strips were brought to land, where they were cut into smaller pieces and put in copper boilers and boiled to train oil (Prestvold, 2001). After impurities in the oil were filtered out, it got placed in casks and was brought back to the Netherlands with the ships at the season's end (Prestvold, 2001). With the change of whaling

strategies in the mid-17th century, Smeerenburg fell out of use and was abandoned by 1660.

By the mid-17th century, climatological changes made it harder for the whales to roam the coastal waters, and they began seeking out food along the outer sides of the drift ice (Joost C. A. Schokkenbroek et al., 2008). The Bowhead Whales also adapted to the presence of whalers in their ecosystems and consequently changed their behavioural pattern (Degroot, 2022). By exploiting their natural adaption to the Arctic -for example the lack of a dorsal fin- they could slip under the ice, hold their breath, and eventually use their strength to break through the surface of the ice Bowheads. In this way they have been able to escape attacks from killer whales (Degroot, 2022). This same behaviour was observed by the whalers and proved effective, as the sea ice could easily sink the ships of the whalers (Degroot, 2022). They were also observed to back away from the ships, sink abruptly to shallows, and remove harpoons by rubbing their bodies against the sea ice (Degroot, 2022).

As the whales moved away from the coast, the Dutch whalers had to transition from bay-whaling to ice-whaling. They would no longer tow the whale to the remote shore; instead, they would be tied to the ship's portside, where the processing would occur. The blubber was cut into smaller pieces and kept in casks, and the baleen was cut from the mouth and hoisted on board. Then it was brought back to the Netherlands, where they had cookeries close to the ports that boiled down the pieces of blubber and turned it into profitable oil (Joost C. A. Schokkenbroek et al., 2008). Therefore it is assumed that most of the cookeries on Spitsbergen were no longer in use as of 1670 (Joost C. A. Schokkenbroek et al., 2008) as all hunting now took place out in the ocean along the edge of the drift ice (Prestvold, 2001) At the same time, the Dutch established contact with the indigenous people along the shores of the Davis Strait and engaged in regular trade of goods. European luxury commodities were exchanged for blubber from seals and whales, along with baleen, tusks from narwhals, and furs (Joost C. A. Schokkenbroek et al., 2008). This lasted until the Kingdom of Denmark took control of Greenland and forbade them to continue the trade (Joost C. A. Schokkenbroek et al., 2008).

Figure 5. Dutch whalers in the Arctic ca. 1700. (Storck, 1700)

The Dutch whaling industry peaked in 1721, with 258 ships with approximately 11,000 whalemen sailing towards Spitsbergen and the Davis Strait. The Dutch supremacy in whaling over the other European nations continued until the end of 18th century (Kruse, 2020; Joost C. A. Schokkenbroek et al., 2008). Every year, the Dutch would bring home between 100 and 200 whales; during the second part of the 17th century, this figure had risen to 1000 to 1500 (Hacquebord, 1985). In the late 18th century, there was a significant decline in the number of whales caught and, subsequently, a decline in the number of ships whaling under the Dutch flag (Joost C. A. Schokkenbroek et al., 2008). In the last quarter of the 18th century, the Fourth Anglo-Dutch War (1780-1784) and the French Napoleonic Wars (1795-1802) paused what little whaling activity was left. By the beginning of the 19th century, the Dutch whaling industry had been reduced to almost nothing (Joost C. A. Schokkenbroek et al., 2008), and the Bowhead Whale population is thought to have been close to extinction (Prestvold, 2001).

During the 19th century, the Dutch attempted several times to re-establish whaling with various results. During a 70-year period, a total of 113 expeditions were undertaken by the Dutch in the Arctic (Schokkenbroek, 2008). They were primarily unsuccessful and rarely managed to bring in any whales. They were, however, occasionally successful in bringing back large quantities of seals. In the 1870s, new technological and methodological advances were made in order to make Dutch whaling a profitable business again, but these attempts were also unsuccessful. Because these voyages brought back so few whales, there isn't much information regarding the species; however, it appears that the majority of them were fin whales (Schokkenbroek, 2008)

2.2 The invisible resource

As discussed, Whales have served as a bountiful resource for coastal communities for millennia. Each individual specimen produced valuable products such as oil, meat, and material for tools. Despite the vast quantities of valuable products they supply, whales have been almost absent from the archaeological record (Charpentier et al., 2022). There are several reasons for this absence. The main reason for the lack of whales in the archaeological record is largely due to how they were processed. Because of their enormous size, the butchering of the whales would have taken place on the beach (Hennius et al., 2022).

Figure 6. Whale bone on display at the Svalbard Museum. Photo by Author.

While the blubber and meat were removed, the bones were large and heavy and would seldom be brought inland intact (Charpentier et al., 2022). When they were brought inland, they were often fragmented or transformed, making them difficult to be identified as whale. The remains left at the beach were then broken down and dispersed by the action of the waves, leaving little or no archaeological traces (Charpentier et al., 2022). Because the majority of the by-products were organic materials that would degrade quickly, the few bones brought back would most

likely be the only by-product from the whales that would survive in significant numbers (Hennius et al., 2022).

It is also particularly unusual to uncover equipment associated with whale exploitation in the archaeological record because whale exploitation did not require any special technology or abilities outside of what was already available (Hennius et al., 2022; Rodrigues et al., 2016). The exception to this is slab-lined pits, which are associated with the rendering of oil from marine mammals (Hennius et al., 2022).

This is not to say that whale bones are entirely absent from the archaeological material. Whaling can be indicated by finds of weaving swords, plaques, cleavers, cutting boards and gaming pieces made out of whale bones. Some organic material has also been discovered; however, it is scarcer than bones (Hennius et al., 2022). In a few cases, baleen has been found in archaeological contexts, i.e., baleen used to fasten the boards in the Oseberg ship (Isaksen, 2017; Hennius et al., 2022). Whale oil itself has not been preserved in the archaeological record. Still, its production can be identified by visible remains of productions such as the slab-lined pits found in Norway (Hennius et al., 2022). However, this lack of whale bones and other related materials in the archaeological record is not incompatible with active whaling. By interpreting a lack of evidence in the archaeological record as a lack of whaling, we risk underestimating the relevance of whales to coastal populations and leaving a gap in our understanding of the past relationship between these giant mammals and humans.

Figure 7. Baleen plates from the collection of Svalbard Museum. Photo by Author.

With little material available in the archaeological record, it is critical to use what is available to try to fill in the gaps in our current understanding of these previous relations. One method is to contextualise the bones that we do have, in terms of their chronology, find locations and thus cultural context. This can be done by radiocarbon dating.

Chapter 3: Whale Selection

The whale taxa chosen are the three species considered to have been important targets during medieval and early modern whaling. The chosen whale species for this study are the North Atlantic Right Whale (*Eubalena glacialis*), Bowhead Whale (*Balaena Mysticetus*), and Gray Whale (*Eschrichtius robustus*). When reservoir-correcting radiocarbon dates for animals, it is important to consider where the animals lived, what they ate, and how long they lived, as these are all factors that can contribute to the result. Since this information can be relevant, as well as providing context for the significance of these whales in history, I have dedicated it to a separate chapter.

3.1 North Atlantic Right whale (*Eubalena glacialis*)

The North Atlantic Right whale (*Eubalena glacialis*) is one of the three right whale species. It is sometimes said to have gotten its name from being the «right whale to hunt» and might be the species that has had one of the most significant influences on the relationship between humans and the sea.

With their large body and high weight, they are amongst the largest of the whales. Their body is uniformly dark in colour with white patches on the belly and chin. Their head is enormous and close to one-third of their body length. Their head has a strongly arched and narrow nostrum, and a bowed lower jaw giving the impression of the whale being upside down (Kraus & Rolland, 2007).

Figure 8. Image of the North Atlantic Right Whale (Eubalena glacialis) (Deedy, 2018)

Around the rostrum, behind the blowholes, over the eyes, on the corners of the chin and variably along the lip and margin of the jaw, thickened patches of skin are found (Kraus & Rolland, 2007). These are callosities and are composed of spikes of columnar epithelial tissue and may look similar to barnacles (Kraus & Rolland, 2007). These callosities are often yellow or cream-coloured due to infestation of whale lice. The functions of the callosities are virtually unknown, but they are unique for each whale and can be used as a means of identification.

The maximum life expectancy of Right whales is essentially unknown. The oldest right whale of known age is estimated to be between 65-70 years old (Philip K. Hamilton et al., 1998; Kraus & Rolland, 2007). Due to evolutionary similarities, this might open the possibility for the Right Whales to be very long-lived on par with the Bowhead Whale (*Balaena mysticetus*). There have been successful studies on ageing other species of whales, e.g. (Boye et al., 2020) and (George et al., 1999), but currently, there is no reliable method to age Right Whales (Kraus & Rolland, 2007).

The migration of the North Atlantic Right Whales is quite different from the migration of other whales. They do not form pods or traverse in larger groups. They are primarily observed in singles or pairs. Larger groups are more likely to develop in areas where they feed and breed. This search for food and mates seems to be the motivation behind the whale's chosen destination (Kraus & Rolland, 2007).

Right Whales mainly feed on calanoid copepods (rice-sized zooplankton) and other small invertebrates, such as smaller copepods, krill, pteropods, and larval barnacles (Jefferson et al., 1996). They feed by swimming continuously with their mouth agape at the surface, also referred to as skim feeding, or at depths down to 200m, filtering the zooplankton that collects on their baleen plates (Kraus & Rolland, 2007).

The North Atlantic Right Whale was among the first species of whale to be commercially hunted (Hennius et al., 2022; van den Hurk, 2020). They were ideal targets due to their large size, which meant large quantities of meat, oil, and baleen (van den Hurk, 2020). They were also a slow-moving species that preferred inshore waters (Aguilar, 1986) making them an easier catch with limited resources such as smaller boats and simpler tools (Hennius et al., 2022). They would move inshore, especially during calving season. As a result, the whalers would target the calves as they were easier targets; this would also lead the mother to come to its rescue, making her an easy kill (Aguilar, 1986). Today the species still hasn't recovered from the over-exploitation by the whaling

industry, and currently, fewer than 400 individuals remain (Frasier et al., 2022). Thus the North Atlantic Right Whale is critically endangered.

3.2 Bowhead Whale (Balaena mysticetus)

The Bowhead whale (*Balaena mysticetus*) has had several names throughout history; Greenland Right Whale, Arctic Whale, and Polar Whale are some. They are in likeness with the North Atlantic Right Whale, a member of the family Balaenidae. They were another of the major targets during the era of commercial whaling. It has also always been the main target of Inuit whalers in the Arctic. Their hunt for the Bowhead Whale is an integral part of their culture and is still taking place today under some regulations (Laugrand & Oosten, 2013).

The Bowhead whale is easily identifiable by its large size and lack of dorsal fin, with a large triangular head that takes up a third of its body, proportionately larger than that of other baleen whales (Ridgeway & Harrison, 1985). The body is black and brown in colour, with well-defined areas of white patterns, making them distinguishable from similar whales such as the right whales (*Eubalena glacialis*) (Rugh & Shelden, 1995). They are large mammals that can weigh up to 100 tons and have flukes reaching 6 metres across. The Bowhead Whales are long-lived cetaceans uniquely adapted to live year-round in the Arctic. This has resulted in them having no dorsal fin and a slow, conservative life-history strategy that, amongst other things, includes incredible longevity (Kovacs et al., 2020). The Bowhead whales may live longer than any other mammal and are believed to be able to live more than a century, as proved when traditional hunting tools were recovered from a modern whale (George et al., 1999).

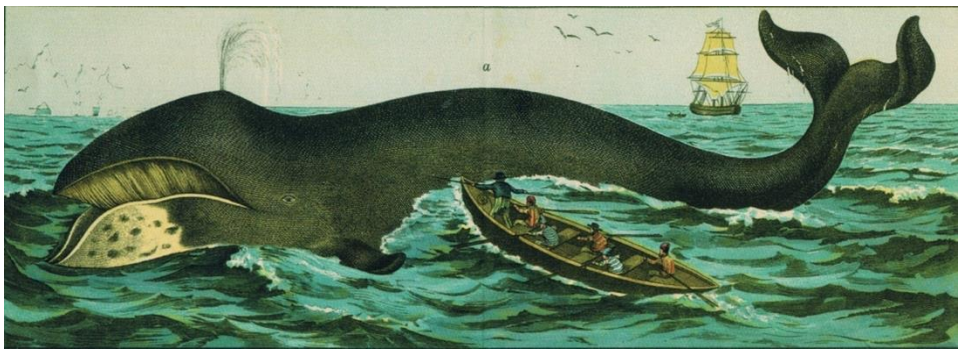

Figure 9. Drawing of a Bowhead Whale (Balaena mysticetus) ca. 1870. (Reproduction) (Wikimedia Commons)

The Bowhead whales around Spitsbergen were hunted to near extinction during the whaling period (Kovacs et al., 2020). Today there are four recognised subpopulations of the Bowhead: Bering - Chukchi - Beaufort Seas; East Canada - West Greenland; East Greenland - Svalbard - Barents Sea; and Okhotsk Sea (IUCN, 2018). The stocks are, for the most part, separated by the sea ice. They spend their lives in and near the sea ice and migrate seasonally to avoid entrapment by the ice and to take advantage of food availability.

Before they were hunted, the East Greenland - Svalbard - Barents Sea population, also referred to as the Spitsbergen stock, was the most extensive stock of Bowheads, numbering somewhere in-between 25 000 to 100 000 whales (Kovacs et al., 2020). Today the remaining population consists of somewhere in-between 50 to 250 mature individuals. They are no longer considered critically endangered but endangered (IUCN, 2018). They would migrate northeast during the spring along the retreating edge of the congregated sea that constitutes the Arctic ice pack (Degroot, 2022). By early April, they reached their feeding grounds in the bays along Svalbard's largest islands that now were clear of large amounts of sea ice (Degroot, 2022). The Bowheads would remain there until the sea ice formed again in the autumn, at which point they would leave for the open ocean.

The Bowhead whale mainly feeds by skimming. They do this by swimming in coordinated formations near the surface through a concentration of zooplankton with their mouth wide open (Ridgway & Harrison, 1989). Mature individuals strain up to nearly two tons of zooplankton through their hundreds of baleen plates at once, some of which reach up to four meters long, the longest of any whale (Rugh & Shelden, 1995). Their diet consists mainly of small to medium-sized zooplankton; with Euphausiids and Copepods comprising the larger part of their diet (65 % and 30 %) (Ridgway & Harrison, 1989). During the winter season, when their regular feeding grounds are covered in sea ice, the bowhead feeds on benthic amphipods near the bottom in shallow areas (Ridgeway & Harrison, 1989; George et al., 1999).

Extensive feeding is necessary for the bowhead to maintain its large body. These giant bodies are an evolutionary adaption to the cold climate of the Arctic and allow for vast energy storage in the form of insulating layers of blubber, and can be more than fifty centimetres thick (Degroot, 2022).

Unfortunately, these adaptations are what made them ideal targets for the whalers. The whalers sought the bowheads for their blubber (oil) and long baleen plates. Due to their incredible weight, they

were slow to escape the boats. In Spitzbergen, European hunting for the bowhead began in the early 1600s. The Dutch quickly dominated the hunt until mid-18th century when the British joined in. By the mid-19th century, it was no longer profitable due to the massive depletion in stock (Ridgeway & Harrison, 1989).

3.3 Gray Whale (Eschrichtius robustus)

The Gray Whale (*Eschrichtius robustus*) can be recognised easily among whale species. The Gray Whale have a distinct body form, almost torpedo-like from above, with a pattern of grey and white splotches. They range in colouration from brownish-grey to light grey (Jefferson et al., 2015). The body is nearly covered with whale lice and barnacles, especially on the head and tail. They have a series of «knuckles» on the dorsal ridge. Gray Whales are among the most coastal of all great whales and live most of their life within tens of kilometres of shore (Jefferson et al., 2015). They are most known for their annual migrations, which is one of the longest migrations known for any mammal. During their migration from winter breeding grounds in Mexico to summer feeding grounds in the Bering-, Chukchi-, and Beaufort seas, they cover 15,000 - 20,000 km (Jefferson et al., 2015). Their lifespan is estimated to be between 50 and 60 years (Alaska gov., 2023).

Figure 10. Illustration of the Gray Whale (Eschrichtius robustus) (NOAA Fisheries, 2023)

Today Gray Whales are only found in the North Pacific Ocean and contiguous seas. There are two populations; one larger eastern Pacific stock and a smaller western North Pacific stock (Jefferson et al., 2015). The specimens sampled in this thesis are from the North Atlantic, where early whalers extirpated the Gray Whale. Since they no longer exist, it is hard to say with certainty where they migrated. In Lindquist's (2000) synthesis of historical texts about the Gray Whale, he concludes that they

most likely migrated between Northwest Africa/ southern Portugal and Iceland. Part of their migration included visiting the English Channel and the southern North Sea for feeding season, with some straying off to the Baltic Sea. Based on the lack of Norwegian accounts concerning the Gray Whale, they most likely travelled to Iceland by a narrow route West of Ireland (Lindquist, 2000).

Amongst the Cetaceans, the Gray Whale is specifically adapted for bottom feeding. They roll on their sides during feeding to suck up the bottom sediments and water in shallow waters. Then they expel out the water and sediment while the prey gets trapped inside of their baleen plates. (Jefferson et al., 2015). Their diet mainly consists of amphipods, decapods, polychaete tube worms, clupeid fish, swarming mysids, kelp, and other algae (Jefferson et al., 2015; Lindquist, 2000). During migration, they also feed by surface skimming on both small fish and shrimp-like mysids (Lindquist, 2000). The Gray Whale's unique feeding habits have contributed to it getting names like «Sandlægja» and «Sandæta» in early Icelandic accounts of the species.

It is difficult to know precisely when the Gray Whale was hunted. Dating of Gray Whale remains indicates that they have been a targeted species all the way back to prehistoric times. During the Basques whaling in European waters, the Gray Whale was most likely one of the target species. With its preference for inshore waters, it must have been an ideal target alongside the North Atlantic Right Whale (*Eubalaena glacialis*). The Gray Whale seems to disappear from mention in European historical sources at the end of the 17th century, indicating that this might be when the species was eradicated entirely from the North Atlantic (Lindquist, 2000).

Chapter 4: Materials and Methods

4.1 Selection of Material

The material for this study was selected based on three pre-determined conditions.

Firstly, the bone had to be from one of the three aforementioned species; North Atlantic Right Whale (*Eubalena glacialis*), Bowhead Whale (*Balaena mysticetus*), and Gray Whale (*Eschrichtius robustus*). Taxonomy was determined by collagen peptide mass fingerprinting (ZooMS) and zoo archaeological (morphological) assessment carried out by Youri van den Hurk (pers comm.). ZooMS cannot yet differentiate within the Balaenidae family down to the species level, which means it cannot differentiate between the North Atlantic Right Whale and Bowhead Whale (Hennius et al., 2022; Buckley et al., 2014; Charpentier et al., 2022). On some of the specimens, however, it was possible to determine the species based on a zooarchaeological analysis.

Secondly, after the collagen extraction was carried out, there had to be enough collagen left to radiocarbon date the specimen after some had been used for ZooMS. Lastly, there had to be enough estimated lipid to potentially date it separately.

20 samples were chosen that initially met these conditions.

Species (ZooMS)	Species	Sampe ID	Context	Country	Context date
Gray whale	Gray Whale	TRa-16922	North Sea		Palaeontological (no clear date)
Gray whale	Gray Whale	TRa-16925	North Sea	North Sea	Palaeontological (no clear date)
Balaenidae	Bowhead Whale	TRa-16980	Uitgeest	Netherland	Late Iron Age - modern
Balaenidae	Balaenidae	TRa-16988	Heiloo Zuiderloo	Netherland	1500-1100 BC
Balaenidae	Balaenidae	TRa-16990	Schagen - 't Noord-Marees	Netherland	AD 1200-1299
Balaenidae	Balaenidae	TRa-16999	Rasquert	Netherland	800 BC - AD 1200 or Post AD 1600*
Balaenidae	Balaenidae	TRa-17000	Oosterbierum	Netherland	800 BC - AD 1200*
Balaenidae	Balaenidae	TRa-17002	Slapershaven, Hoorn	Netherland	Unknown
Balaenidae	Bowhead Whale	TRa-17010	Persingen	Netherland	Post AD 1600
Balaenidae	Bowhead Whale	TRa-17013	Smeerenburg, Svalbard	Norway	Post AD 1600
Balaenidae	Prob. North Atlantic Right Whale	TRa-17027	Holwerd	Netherland	800 BC - AD 1200*
Balaenidae	Bowhead Whale	TRa-17031	Rasquert	Netherland	Post AD 1600*
Balaenidae	Bowhead Whale	TRa-17092	Winsum	Netherland	Post AD 1600*
Balaenidae	Prob. North Atlantic Right Whale	TRa-17134	Province of Friesland	Netherland	800 BC - AD 1200*
Balaenidae	Prob. North Atlantic Right Whale	TRa-17814	Trondheim	Norway	Late Iron Age - Medieval
Balaenidae	Balaenidae	TRa-17847	Walstraat, Vlissingen	Netherland	Post AD 1600
Balaenidae	Bowhead Whale	TRa-17869	Province of Flevoland	Netherland	Post AD 1600
Balaenidae	Prob. North Atlantic Right Whale	TRa-18015	Uddevalla	Sweden	Unknown
Balaenidae	North Atlantic Right Whale	TRa-18032	Gobiendes, Colunga	Spain	Likely post AD 1575
Balaenidae	Balaenidae	Tra-16995	Velsen A9 / A22	Netherland	1300-1100 BC

*Table 1. Table of alll the bones sampled. The second species column is based on the zoo archaeological assessment. Some context dates are marked with the * symbol. These are based the context of their sample but have significant uncertainty due to the particular type*

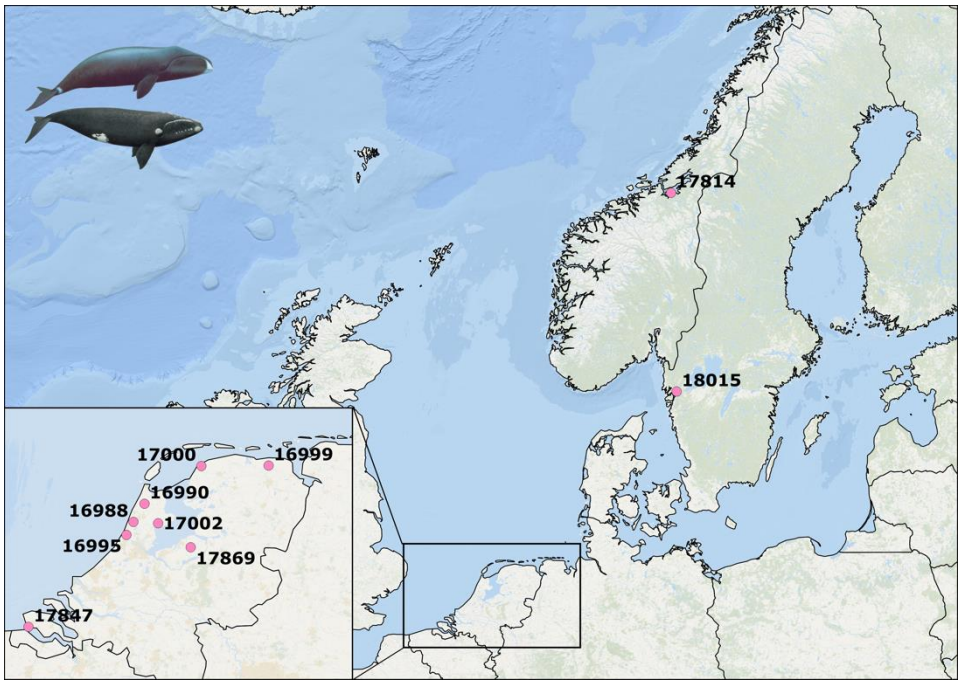

Figure 11. Map showing where the Balaenidae samples are collected from. Map by Author.

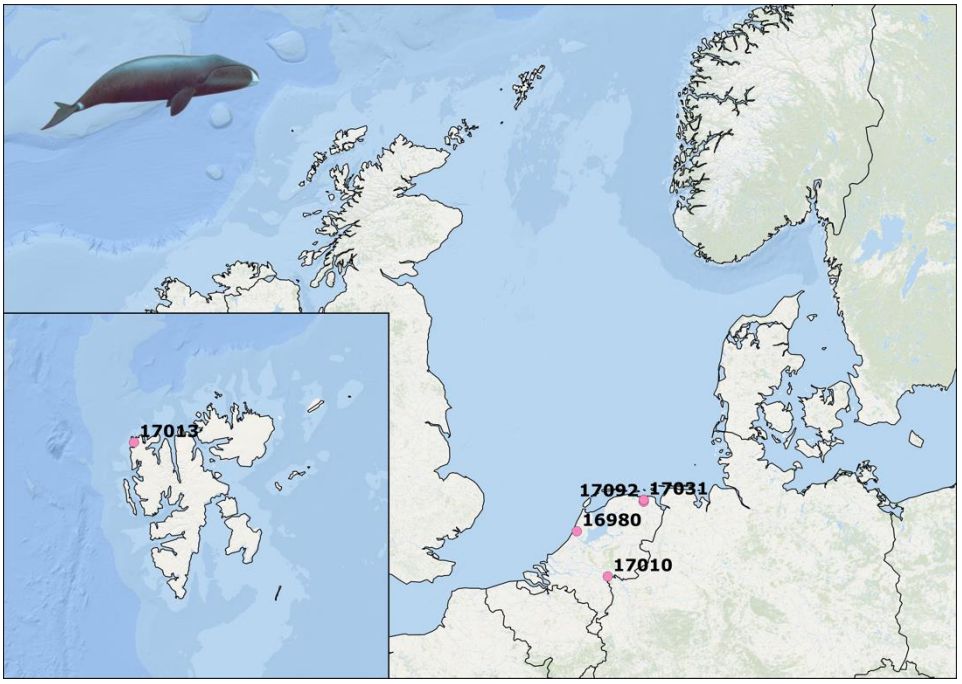

Figure 13. Map showing where the Bowhead Whale (Balaena mysticetus) are collected. Map by Author.

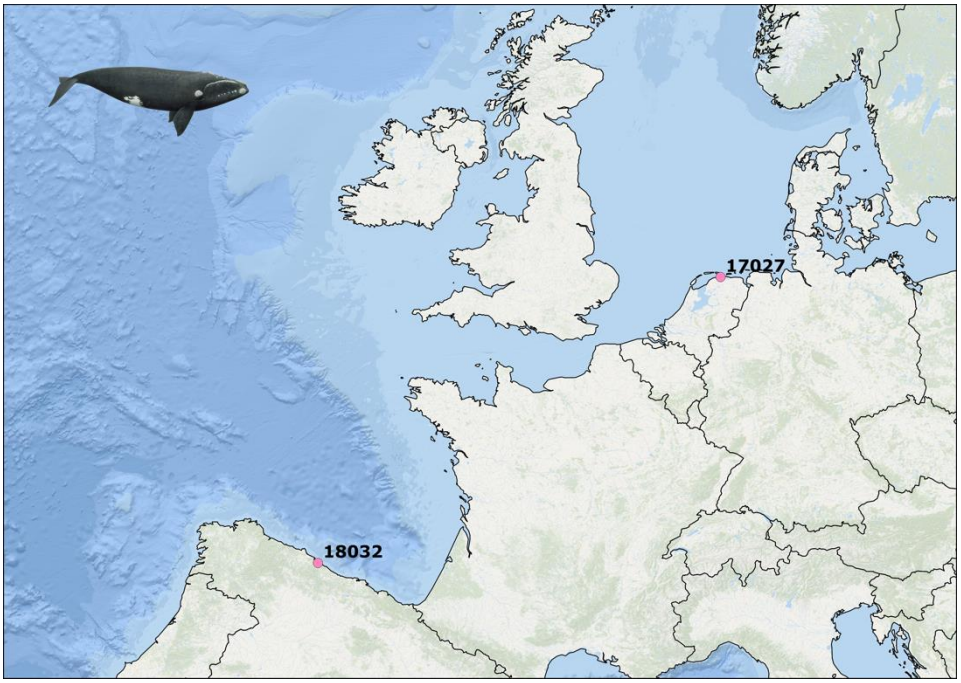

Figure 12. Map showing where the North Atlantic Right Whale (Eubalena glacialis) samples are collected from. Map by Author.

The Gray Whale samples were found in the North Sea and have no known collection site.

4.2 Methods

The radiocarbon dating was done at the National Laboratory for Age Determination in Trondheim at the 1 MV AMS system (Seiler et al., 2019).

The pre-treatment protocol for the collagen was developed by Youri van den Hurk and Marie-Josée Nadeau with inputs from James Barrett. The lipid pre-treatment was developed by myself and Marie-Josée Nadeau. The collagen and lipid were extracted from the samples by myself.

The dates were calibrated using the marine radiocarbon age calibration curve Marine20 by using OxCal v4.4.4 program (Ramsey, 2021). The ΔR was calculated by using OxCal v4.4.4 program (Ramsey, 2021). The FTIR spectroscopy was done at the Conservation lab of the NTNU University Museum with a Perkin-Elmer Spectrum 400 spectrometer with a Universal ATR diamond crystal device using the Spectrum software.

The following chapters will go through these methods in detail.

4.2.1 Bone pre-treatment and collagen extraction

The collagen was extracted from the bones by using a modified version (van den Hurk, pers comm.) of the standard procedure for bone preparation for the National Laboratory for Age Determination in Trondheim (Seiler et al., 2019), with the addition of a lipid extraction step (See Appendix 1).

Sample Preparation

The bone samples were first crushed into smaller pieces (≈ 0.5 grams) and underwent visual inspection to see if the bone was treated with glue and if any carbon contaminants needed to be removed.

Following that, the samples were cleaned in an ultrasonic bath with 18.2 MΩ-cm ultrapure water (Type 1).

They were cleaned three times for five minutes, or until all visible dirt was removed and the water seemed clear. It is possible that with some samples that had been crushed into a fine powder, parts of the specimen were mixed in with the sediment, and higher care needed to be taken to avoid removing bone powder along with the contamination. After washing, the sample was left to air. After the evaporation, the sample

was re-weighed to assess the sample weight got in the washing and to determine the sample mass for later calculations.

Lipid extraction

After sufficiently preparing the sample, a dichloromethane/methanol (2:1) solvent was added to remove the lipid from the bone (Cequier-Sánchez et al., 2008). The solvent was added until the sample was completely submerged, and then it was ultrasonicated for fifteen minutes. The lipid was then extracted with a pipette into another tube. This process was repeated three times or until there was no longer any discolouration in the solution.

Between each ultrasonication, the solution was changed, and the temperature of the bath lowered as dichloromethane/methanol will boil around 40 °C (Kim et al., 2023).

Then the tubes with the remaining bone sample and lipid were set aside to allow the dichloromethane/methanol to evaporate completely.

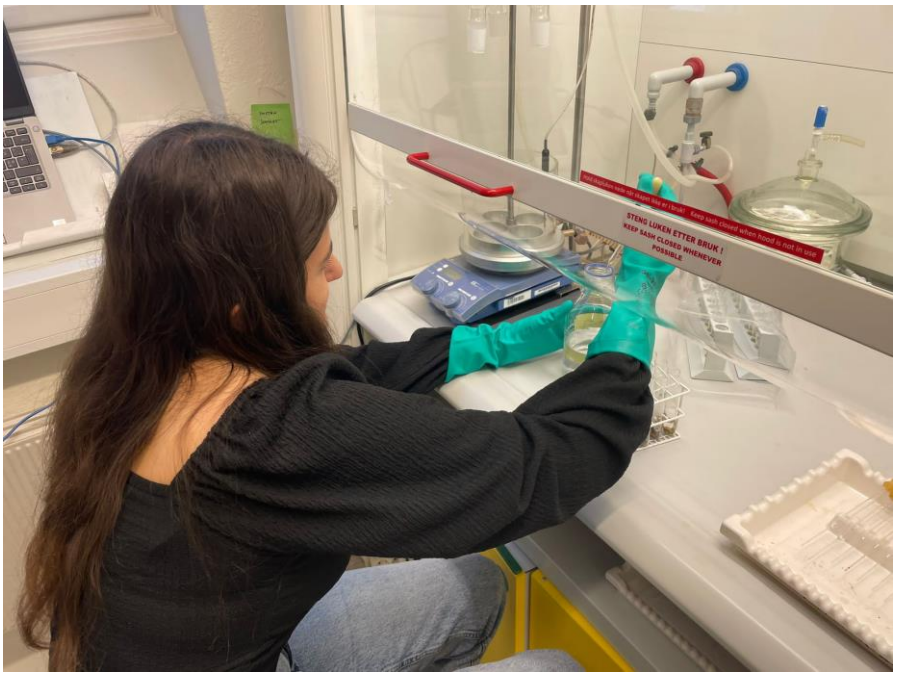

Figure 14. Lipid extraction in progress. Photo: Youri van den Hurk

Collagen extraction

After the sample had finished drying, it was demineralised overnight using 2.44 M HCl (50ml solution per 100 mg of bone) within a vacuum desiccator at room temperature. After the solution was added, the tubes were covered with Parafilm with a small hole punctured at the top and then put in the desiccator. To avoid foaming at the top of the tube, it had to be pumped slowly.

To accelerate the demineralisation, it needed to be pumped every fifteen minutes. After two hours, or if the bubbling stopped, the pH was checked. If the pH was >1, 2-3 drops of concentrated acid were added, and any pieces of bone stuck to the tubes' walls got pushed back into the acid. When the bubbling stopped, the sample stay 30 minutes before rechecking. If the bubbling then continues, the previous step was repeated.

Figure 15. Samples in the desiccator. Photo by Author.

The acid was then removed from the test tube by carefully using a pipette. A little liquid was left above the sample on the more powdered samples to avoid sucking up any of the specimen. The sample was then washed with ultrapure water 3 times (or as many as necessary) until the pH had reached 3 or above. Then around 4ml of 0.5 % NaOH was added to the sample. The sample was then left between 2-4 hours (never more than four). This was in order to remove humic acids. After this, the sample was washed with ultrapure water until the pH measured <10.

To remove any atmospheric CO_2, absorbed during the NaOH step. 5 ml of 1.22 M HCl was added at room temperature for 1-2 hours. The final pH was approximately 1. Then the sample was rinsed with ultrapure water until a pH of 3.0 ± 0.2 was achieved (if it exceeded 3, more HCl was added to decrease the pH). When the sample reached the ideal pH with around 5mL of liquid, it was put in the oven at 70 °C overnight in order to hydrolyse the collagen to gelatine.

The gelatine was then filtered using a pre-baked (850 °C for 6 hours) quartz aerosol filter (Merck Millipore, AQFA04700, 99.998 % for

0.3 µm particles). The filtrated gelatine was frozen first in the freezer and then freeze-dried, leaving collagen ready to be dated.

4.2.2 Development of Lipid pre-treatment method

The lipid extracted went through additional pre-treatment before dating. To ensure that the best possible lipid yield for radiocarbon dating would be achieved, several methods for filtering fat were tested before treating the actual lipid extracted during the collagen pre-treatment process. In replacement of whale lipids, experimentation used fat from bacon instead.

Filtrating through quartz filters

The fat was placed in pre-weighed tubes. Dichloromethane/Methanol (2:1) was added until the fat was covered. The tubes were put in the ultrasonic bath for 15 minutes. This caused the fat to liquefy, making it easier to transfer from the tube. The fat was then pipette-filtered through a pre-baked (850 °C for 6 hours) quartz filter (Merck Millipore, AQFA04700, 99.998 % for 0.3 µm particles). The filters were then flushed with the Dichloromethane/Methanol solution to ensure that as much fat as possible was filtered through. When the Dichloromethane/Methanol had finished evaporating the tube got re-weighed to calculate how much fat had gone through.

The expectation was that most of the fat would filter through, leaving any dirt and/or contaminant behind in the filter. Alas, the results showed, on average, that less than ≈20% of the original sample weight got through the filter, while 70-80% got stuck in the filter. There was also some visible fat residue on the sides of the funnels that held the filters.

For the next attempt, the same method was tested. However, adding the fat onto the filters was done with more haste so that the solution would remain warm after the sonication. It was pipetted more centrally and slowly onto the filter to avoid too much hitting the funnel sides, a more thorough rinse with Dichloromethane/Methanol was performed afterwards. This resulted in a slightly better yield (≈25 %) but a more extensive amount of the sample got stuck in the pipette. Yet still, more than half of the sample was stuck in the filter. This would not have been an issue on an ordinary sample, considering that most of the original weight might be due to dirt or other contaminants. However, these samples were thought to be primarily pure fat, which might mean that on an ordinary sample, most of the lipids would also get stuck in the

filter. Therefore a method for extracting the fat out of the filters without also removing the contaminants was needed.

The «mini-soxhlet»

To try and solve this problem, the first choice was using a soxhlet to extract the fat from the filters. It is an extraction method widely used for many types of solid samples and is the standard technique for extracting samples for analyses from solid samples (Zygler et al., 2012). The soxhlet extraction is a complicated process, but the principle is that heat and condensation of the solvent allow for repeated extraction from the sample. We were concerned that because the fat was challenging to remove from tubes and filters, so it would be even more difficult to remove from the soxhlet equipment. Therefore we opted not to use a soxhlet.

The goal became to achieve the extraction of lipid provided by a soxhlet, but without having to deal with the extraction of the lipid from the soxhlet afterwards. We also needed something of a smaller size. Therefore, we created what we called the «mini-Soxhlet». The «mini-Soxhlet» was made up of two components. The first part was made by twisting old silicone tubes around the top of the test tubes and connecting it to the cold water tap in the sink. This allowed for a continuous cold water flow around the top of the test tubes. In the test tubes, the filter with the lipid stuck in it was soaked in Dichloromethane/Methanol solution, and the top was plugged with a silicone plug. The second part was a heating source. Since Dichloromethane has quite a low boiling point, around 39-40 °C (Kim et al., 2023), not much heat was needed. We ended up putting the wrapped test tubes into the ultrasonic bath with the heat function set to a little higher than the boiling point for Dichloromethane/Methanol. The

combination of the cooling tubes and the bath's heat allowed the solution to boil and quickly condense again.

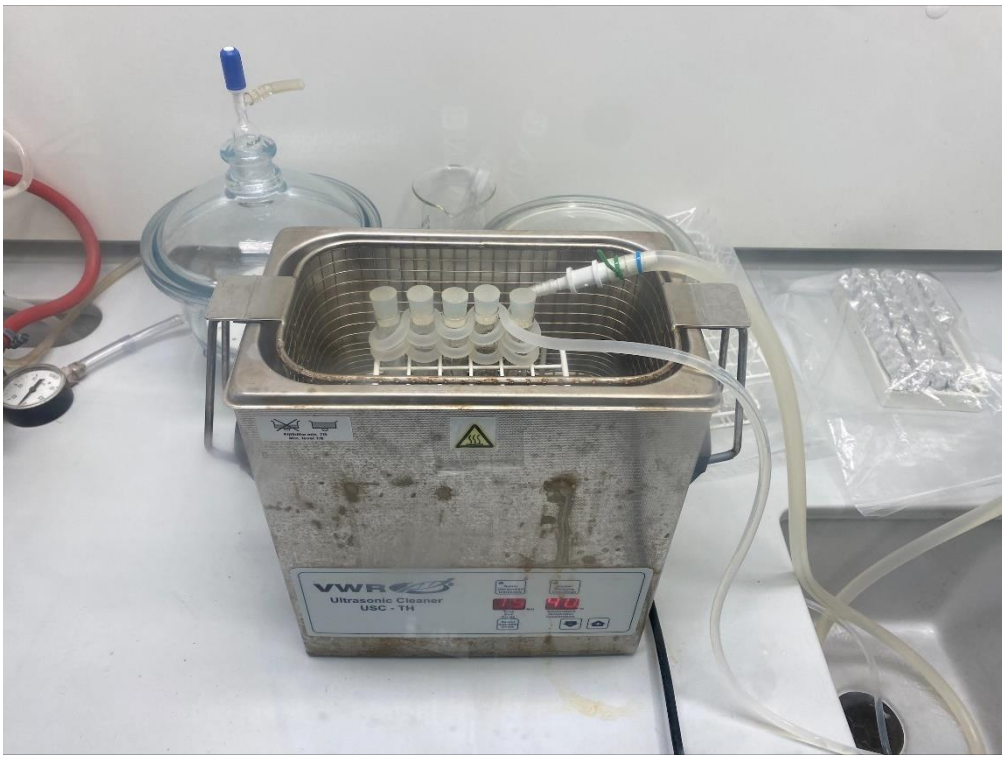

Figure 16. The "mini-soxhlet" in action. The silicone tubes are attached to the cold water faucet to the right. Photo by Author.

To keep the filters in place in the test tubes and to ensure that none of the contaminants on the filters leaked into the solution, the filters were folded into small «parcels» that were kept in place by sterilised paperclips, which had been pre-treated with Dichloromethane/Methanol, Acetone, and Ethanol. After several attempts, this method consistently resulted in ≈80% fat yield.

To easily remove the fat from the tube for dating, another pre-baked quartz filter was placed in the solution right after the paperclip parcel was removed. This managed to soak up most of the fat while the Dichloromethane/Methanol evaporated.

After all the Dichloromethane/Methanol had evaporated, the filters were weighed to make sure there was enough fat to possibly date. Then the standard procedure for combustion and cracking was tested. The procedures of combustion and cracking are methods used to extract the Carbon from the sample. They were sufficient in extracting enough Carbon from the filters.

Thus, we had a pre-treatment that seemed to be efficient in filtering the sample to remove contaminants and extract enough carbon from the lipid so it would be possible to radiocarbon date.

4.2.3 Lipid pre-treatment

The end product of all the testing with fat described in the previous sub-chapter resulted in the lipid pre-treatment that was used for all the archaeological samples (See Appendix 2).

Lipid filtration

The lipid was first extracted by adding Dichlormethane/Methanol and ultrasonication for 15 minutes. Then the lipid was filtered through a pre-baked (850 °C for 6 hours) quartz filter (Merck Millipore, AQFA04700, 99.998 % for 0.3 µm particles). The filter was then dried in the oven overnight at 70 °C while the tubes were left in the fume hood.

The dried filters were folded into small packets, which were held together by a sterilised paperclip and then put in the corresponding tube used during the first filtering. The Dichloromethane/Methanol solvent was then added until the filter was covered entirely. The tubes were then added to the «mini soxhlet» at 41 °C for approximately one hour. After one hour, the paperclip with the filter was removed, and a new pre-baked quartz filter was added to soak up all the lipids while the Dichloromethane/Methanol evaporated.

Combustion and Cracking

The filters that showed a high enough weight (>1mg) for possible dating were placed in a pre-baked (850 °C for 6 hours) quartz tube (outer diameter: 9 mm, inner diameter: 7 mm, length: 180 mm. Multi-Lab Ltd, Newcastle upon Tyne, England) filled with 500 mg CuO pellets and a piece of silver (ca 100 mg Ag). The filled tubes were then pumped until the pressure was below $5 \cdot 10^{-5}$ mbar and sealed using a propane-oxygen flame. The sealed tubes were placed in ceramic (Sillimantin 60) tubes inside the oven, where they were combusted at 850 °C for 6 hours. For the cracking, the samples followed the standard procedure for cracking and outtake of Carbon at the National Laboratory for Age Determination in Trondheim. (see Appendix 3)

4.2.4 ΔR Correction of Bowhead Whale (*Balaena mysticetus)* Samples

In this project, the method used for determining ΔR for one of the specimens is related to the method of using known age samples. Although the precise death age of the specimen is unknown, the site age is known, making it possible to establish a maximum and minimum age.

To establish a ΔR value for the Bowhead Whale samples, the sample from Smeerenburg (TRa-17013) was used. Smeerenburg was mainly used/occupied between 1619-1660 CE (Hacquebord, 1985). Since this is such a short time span, it allows for estimating the ΔR of this sample.

Firstly, the boundaries of the age range of the sample were introduced with the use of the C_Date function in OxCal. This limited the dating results to staying between 1619-1660 CE. This means that when adding potential delta corrections to the dating result for TRa-17013, it will consider how well it agrees with the dating result within the limitations specified (See Appendix 4).

It would be ideal to establish one universal ΔR value which could be applied to all, but that is difficult to do as multiple values can be applied and shown as correct. To find a starting point, different already established ΔR values from the Marine20 database (Reimer & Reimer, 2001) were tested until an agreement of 60% or more (a good agreement) was achieved. A good agreement means that the assumptions entered (in this case the delta corrections) agree with the dating results. The corrections tested were from Mangerud, J., 1972, and Mangerud, J. & Gulliksen, S. 1975, as they were all from the same geographical area as TRa-17013, even though these literature values were obtained on shells rather than marine mammals. The objective was to establish a starting point; therefore, the exact numbers used made little difference. One could have just as well used random values.

When several values gave good agreements, I experimented with different intervals to see how much I could extend and narrow the correction while still maintaining a good agreement. This is in order to delineate when the corrections no longer agreed with the known dates of the TRa-17013. The result was a ΔR expressed as the interval (-225, -20). This interval includes all the ΔR values that maintained a good agreement with the TRa-17013 ^{14}C Age. Thus, while this one sample does not allow for a precise calculation of a single ΔR value, we know that possible ΔR values for TRa-17013 range from -225 to -20 ^{14}C years. Afterwards, the interval was tested against the other Bowhead ^{14}C ages

to ensure that the agreement stayed good on all of them and that the delta correction could be applied to all the samples.

4.2.5 FTIR

Fourier Transform Infrared (FTIR) spectroscopy was used on the residue left from the test tubes the lipid was kept in before filtering to see if there were any contaminants we could identify. FTIR spectroscopy is a biophysical characterisation technique widely used in material analysis (Liu et al., 2013). It identifies chemicals based on the interaction of molecules with electromagnetic radiation in the mid-infrared region (Franca & Oliveira, 2022).

The residue that was left in the original lipid tubes was carefully scraped out with a sterilised spatula and placed on the scanner of the machine. The results were then imported to the Spectragryph program and compared to the IRUG.org database as well as some spectrums from the literature.

Chapter 5: Results

5.1 Collagen Results

Species (ZooMS)	Species	Sample ID	Context	Country	Context date	14C Age (rounded)	Calibrated Age Ranges	C content % by weight
Gray whale	Gray Whale	TRa-16922	North Sea		Palaeontological (no clear date)	4600 ± 40	68.3% probability 2772BC (68.3%) 2569BC 95.4% probability 2857BC (95.4%) 2488BC	17
Gray whale	Gray Whale	TRa-16922	North Sea		Palaeontological (no clear date)	4640 ± 35	68.3% probability 2832BC (68.3%) 2647BC 95.4% probability 2886BC (95.4%) 2553BC	17
Gray whale	Gray Whale	*TRa-16922 avera*	North Sea	North Sea	Palaeontological (no clear date)	*4620 ± 35*	*68.3% probability 2811BC (68.3%) 2620BC 95.4% probability 2872BC (95.4%) 2542BC X2-Test: df=1 T=0.6(5% 3.8)*	
Gray whale	Gray Whale	TRa-16925	North Sea	North Sea	Palaeontological (no clear date)	4305 ± 20	68.3% probability 2372BC (68.3%) 2198BC 95.4% probability 2451BC (95.4%) 2129BC	45
Balaenidae	Bowhead Whale	TRa-16980	Uitgeest	Netherland	Late Iron Age - modern	565 ± 15	68.3% probability 1882AD (68.3%) 1950AD 95.4% probability 1918AD (95.4%) 1950AD	46
Balaenidae	Balaenidae	TRa-16988	Heiloo Zuiderloo	Netherland	1500-1100 BC	3610 ± 20	68.3% probability 1464BC (68.3%) 1321BC 95.4% probability 1525BC (95.4%) 1249BC	39
Balaenidae	Balaenidae	TRa-16990	Schagen - 't Noord-Marees	Netherland	AD 1200-1299	1175 ± 15	68.3% probability 1312AD (68.3%) 1413AD 95.4% probability 1270AD (95.4%) 1464AD	42
Balaenidae	Balaenidae	TRa-16999	Rasquert	Netherland	800 BC - AD 1200 or Post AD 1600*	1685 ± 20	68.3% probability 796AD (68.3%) 942AD 95.4% probability 726AD (95.4%) 1006AD	47
Balaenidae	Balaenidae	TRa-17000	Oosterbierum	Netherland	800 BC - AD 1200*	1520 ± 15	68.3% probability 985AD (68.3%) 1121AD 95.4% probability 907AD (95.4%) 1180AD	45
Balaenidae	Balaenidae	TRa-17002	Slapershaven, Hoorn	Netherland	Unknown	675 ± 20	68.3% probability 1734AD (68.3%) 1898AD 95.4% probability 1696AD (95.4%) 1950AD	33
Balaenidae	Bowhead Whale	TRa-17010	Persingen	Netherland	Post AD 1600	575 ± 20	68.3% probability 1876AD (68.3%) 1950AD 95.4% probability 1919AD (95.4%) 1950AD	44
Balaenidae	Bowhead Whale	TRa-17013	Smeerenburg, Svalbard	Norway	Post AD 1600	735 ± 20	68.3% probability 1676AD (68.3%) 1826AD 95.4% probability 1632AD (95.4%) 1935AD	44
Balaenidae	Prob. North Atlantic Right Whale	TRa-17027	Helwerd	Netherland	800 BC - AD 1200 *	1800 ± 15	68.3% probability 681AD (68.3%) 805AD 95.4% probability 646AD (95.4%) 887AD	40
Balaenidae	Bowhead Whale	TRa-17031	Rasquert	Netherland	Post AD 1600*	625 ± 15	68.3% probability 1817AD (68.3%) 1950AD 95.4% probability 1869AD (95.4%) 1950AD	45
Balaenidae	Bowhead Whale	TRa-17092	Winsum	Netherland	Post AD 1600*	600 ± 15	68.3% probability 1854AD (68.3%) 1950AD 95.4% probability 1921AD (95.4%) 1950AD	40
Balaenidae	Prob. North Atlantic Right Whale	TRa-17134	Province of Fricsland	Netherland	800 BC - AD 1200*	2000 ± 15	68.3% probability 490AD (68.3%) 623AD 95.4% probability 430AD (95.4%) 669AD	38
Balaenidae	Prob. North Atlantic Right Whale	TRa-17814	Trondheim	Norway	Late Iron Age - Medieval	1410 ± 15	68.3% probability 1091AD (68.3%) 1225AD 95.4% probability 1039AD (95.4%) 1277AD	35
Balaenidae	Balaenidae	TRa-17847	Walstraat, Vlissingen	Netherland	Post AD 1600	725 ± 15	68.3% probability 1687AD (68.3%) 1836AD 95.4% probability 1650AD (95.4%) 1932AD	45
Balaenidae	Bowhead Whale	TRa-17869	Province of Flevoland	Netherland	Post AD 1600	590 ± 30	68.3% probability 1858AD (68.3%) 1950AD 95.4% probability 1918AD (95.4%) 1950AD	31
Balaenidae	Prob. North Atlantic Right Whale	TRa-18015	Uddevalla	Sweden	Unknown	735 ± 15	68.3% probability 1679AD (68.3%) 1821AD 95.4% probability 1631AD (95.4%) 1927AD	43
Balaenidae	North Atlantic Right Whale	TRa-18032	Gobiendes, Colunga	Spain	Likely post AD 1575	780 ± 20	68.3% probability 1647AD (68.3%) 1799AD 95.4% probability 1550AD (95.4%) 1866AD	40
Balaenidae	Balaenidae	Tra-16995	Velsen A9 / A22	Netherland	1300 - 1100 BC	*Failed*	*Failed*	*Failed*

*Table 2. Results table showing both Uncalibrated and Calibrated dates shown. Some context dates are marked with the * symbol. These are based on the context of their sample, but have significant uncertainty due to the particular type of context.*

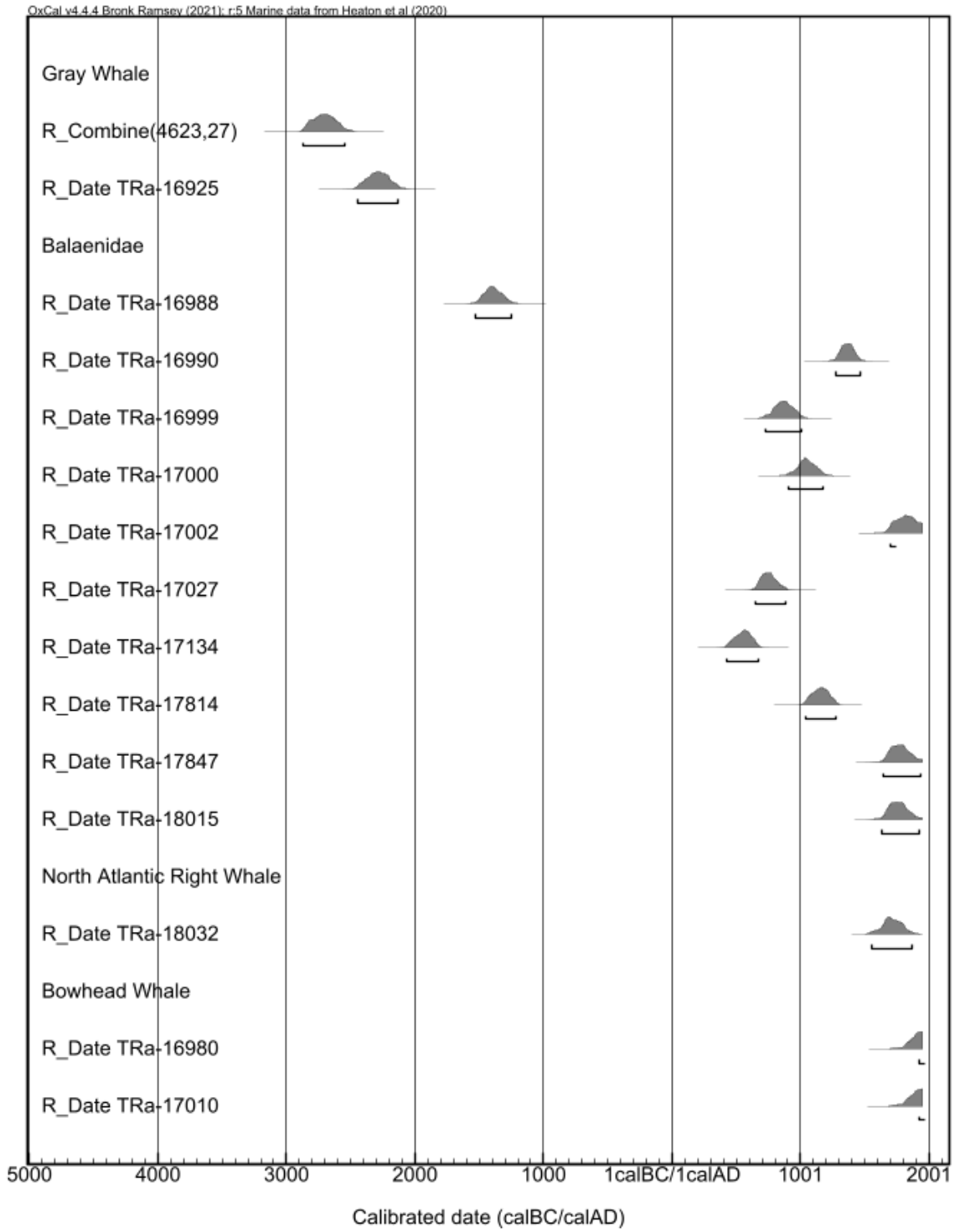

Figure 17. Calibrated Collagen dates modelled in OxCal v4.4.4 program with the Marine20 calibration curve. R_Combine is the average calibration of TRa-16922. No ΔR correction is used on any of the dates modelled.

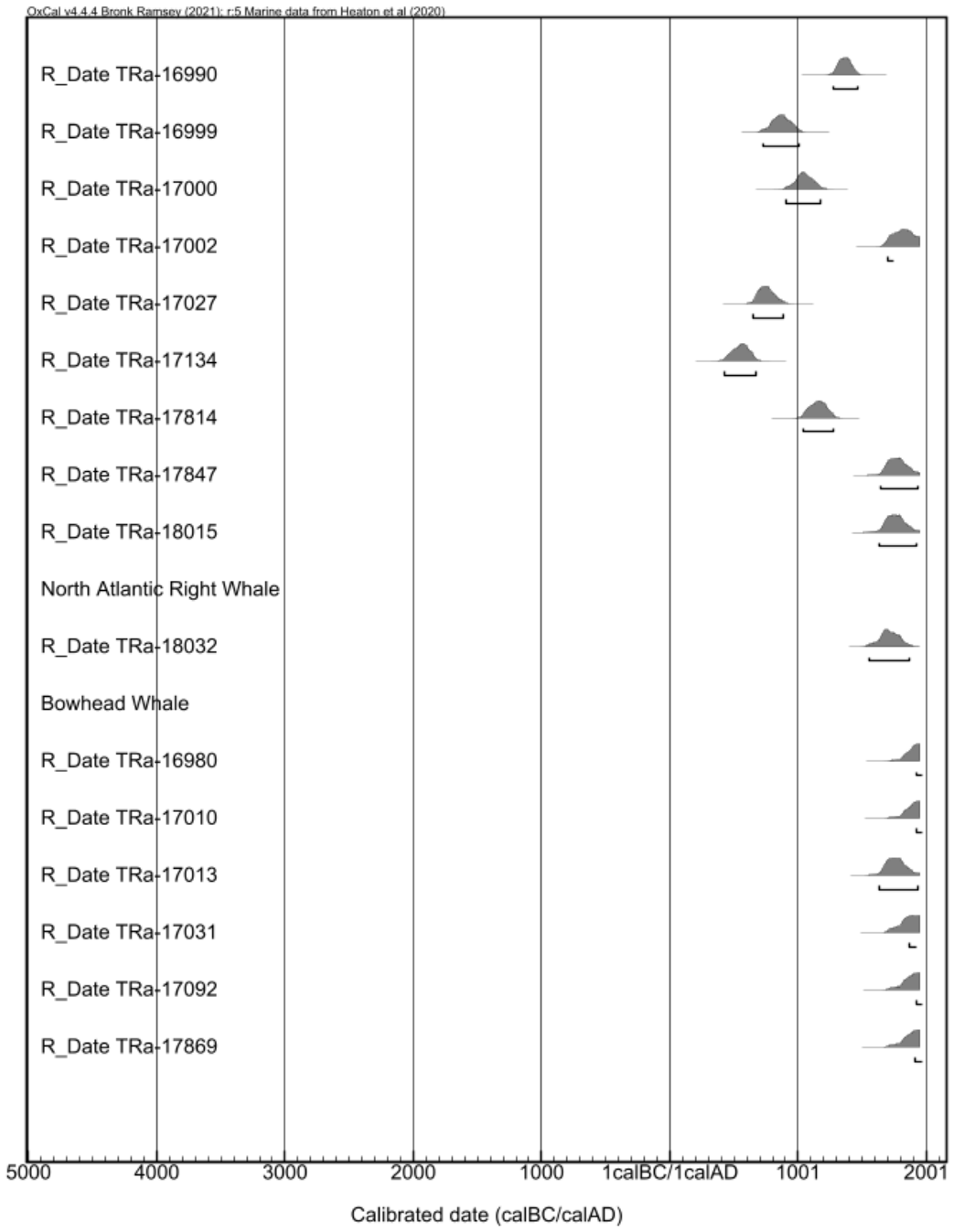

Figure 18. Calibrated Collagen dates modelled in OxCal v4.4.4 program with the Marine20 calibration curve. R_Combine is the average calibration of TRa-16922. No ΔR correction is used on any of the dates modelled.

5.1.1 ΔR calibration of the other Balaenidae and Gray Whale samples

The primary intent for including the North Atlantic Right Whale / Balaenidae and Gray Whale samples was to compare the difference in dates in the collagen and lipid. As they are not samples of known age, it is not possible to calculate a species-specific ΔR. Using an already established ΔR for the area the samples originated from is also not a viable option. The whales are too migratory for a location-based ΔR to be valid. Furthermore, the bones could have been brought back with the whalers and had no relation to their location. Hopefully, the collagen dates can be used in future research when a species-specific ΔR for the species are available.

As there is a possibility for some of the Balaenidae to be Bowheads, the correction could, in theory, be applied to these as well. I have decided against doing that here since there is still a possibility that they are not Bowheads. Additionally, any analysis based on the adjusted dates could potentially be inaccurate.

5.2 ΔR calibrated Bowhead Whale Dates

Species (ZooMS)	Species	Sample ID	Context	Country	Context date	14C Age (rounded)	Calibrated Age Ranges	ΔR Corrected Age Ranges
Balaenidae	Bowhead Whale	TRa-16980	Uitgeest	Netherland	Late Iron Age - modern	565 ± 15	68.3% probability 1882AD (68.3%) 1950AD 95.4% probability 1918AD (95.4%) 1950AD	95.4% probability 1671 (95.4%) 1953 AD
Balaenidae	Bowhead Whale	TRa-17010	Persingen	Netherland	Post AD 1600	575 ± 20	68.3% probability 1876AD (68.3%) 1950AD 95.4% probability 1919AD (95.4%) 1950AD	95.4% probability 1660 (95.4%) 1953 AD
Balaenidae	Bowhead Whale	TRa-17013	Smeerenburg, Svalbard	Norway	Post AD 1600	735 ± 20	68.3% probability 1676AD (68.3%) 1826AD 95.4% probability 1632AD (95.4%) 1935AD	95.4% probability 1619 (95.4%) 1659 AD
Balaenidae	Bowhead Whale	TRa-17031	Rasquert	Netherland	Post AD 1600*	625 ± 15	68.3% probability 1817AD (68.3%) 1950AD 95.4% probability 1869AD (95.4%) 1950AD	95.4% probability 1587 (95.4%) 1953 AD
Balaenidae	Bowhead Whale	TRa-17092	Winsum	Netherland	Post AD 1600*	600 ± 15	68.3% probability 1854AD (68.3%) 1950AD 95.4% probability 1921AD (95.4%) 1950AD	95.4% probability 1633 (95.4%) 1953 AD
Balaenidae	Bowhead Whale	TRa-17869	Province of Flevoland	Netherland	Post AD 1600	590 ± 30	68.3% probability 1858AD (68.3%) 1950AD 95.4% probability 1918AD (95.4%) 1950AD	95.4% probability 1638 (95.4%) 1953 AD

*Table 3. Results table showing Uncalibrated, Calibrated (Marine20), and ΔR corrected dates shown. Some context dates are marked with the * symbol. These are based on the context of the sample, but have significant uncertainty due to the particular type of*

The ΔR for the Bowhead Whale (*Balaena Mysticetus*) was calculated to lie in the interval (-225, -20). When this correction and the Marine20 calibration curve were applied to the other Bowhead samples, the dates corresponded with the broad expected dates based on the archaeological context. Therefore, it seems that it is still possible to use the Marine20 curve on Bowhead samples, even though they are a species living outside the recommended latitudinal range. When using a uniform ΔR distribution, expressed as U(-225, -20) in OxCal, or when using a normal ΔR distribution, N(-110, 50) in OxCal, the results had little to no variation.

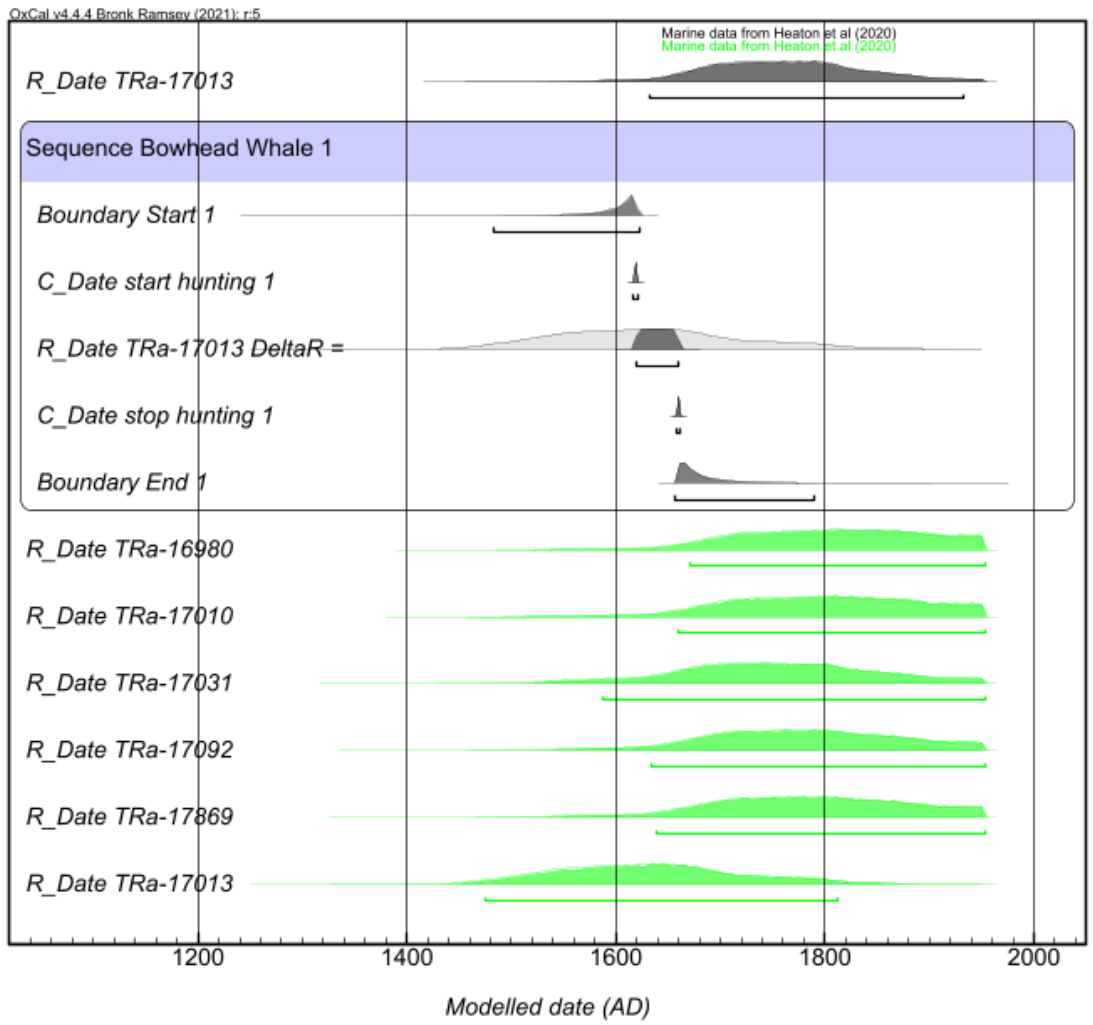

Figure 19. ΔR Calibrated dates for the Bowhead Whale samples modelled in OxCal v4.4.4 program.

5.2 Lipid results

After pre-treatment, only 7 of the 20 samples had a high enough (>1mg) weight for potential dating.

Sample Id	Fraction	14C Age (rounded)	% C	mgC
TRa-16980	Lipid	26600 ± 300	48.5	0.97
TRa-17010	Lipid	25490 +120/-110 BP	52.2	2.66
TRa-17027	Lipid	4090 ± 120	10.8	0.13
TRa-17031	Lipid	6320 ± 45	27.6	0.58
TRa-17092	Lipid	12390 ± 100	31.2	0.50
TRa-17847	Lipid	16600 ± 200	30	0.39
TRa-18032	Lipid	20580 ± 60	50.9	3.36

Table 4. Results table showing the radiocarbon dates of the lipid samples. The dates are significantly older than the collagen dates. The %C is also relatively low. This indicates that these results are incorrect.

The lipids dated much older than their respective collagen dates (see Table 2). That, together with the low %C, indicates that most of the sample was not, in fact, lipid at all. What got dated was most likely contamination. Contamination of the solvent seems improbable as the Dichloromethane/Methanol got sufficient time to evaporate before dating, and a similar protocol had previously been used at the lab for known age samples (Nadeau, pers comm.). It did not result in significant age differences as for these samples. To identify what possibly contaminated the samples, a review of the residue left in the test tubes the lipid was kept in before filtering was done with Fourier Transform Infrared (FTIR) spectroscopy. There were only five of the tubes that had enough residue left.

5.2.1 FTIR spectra of the Lipid samples

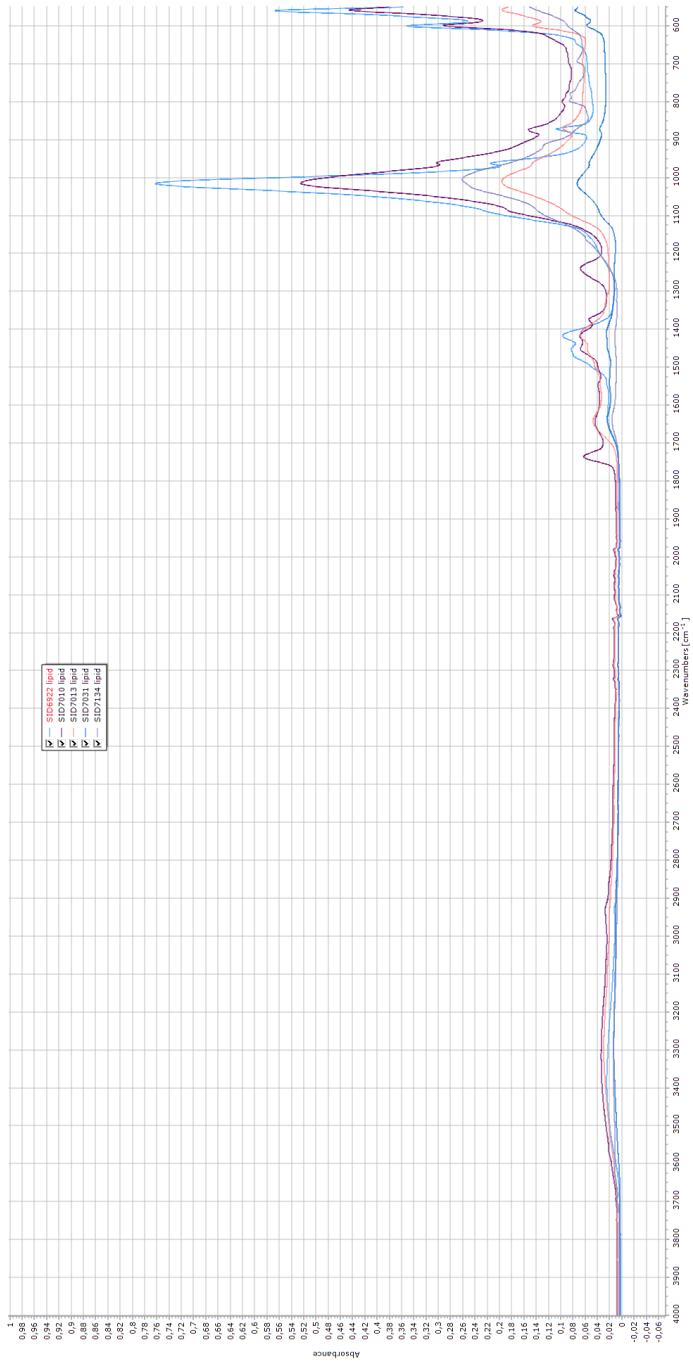

Figure 20. FTIR spectra results of the lipid samples; TRa-16922, TRa-17010, TRa-17013, TRa-17031, and TRa-17134. Lipids usually have an absoption peak around 3000cm-1. The peaks of the samples analysed here match measurements done of different sediments. This makes it unlikely that there were any lipids at all in the samples. Figure made with Spectragryph by Author.

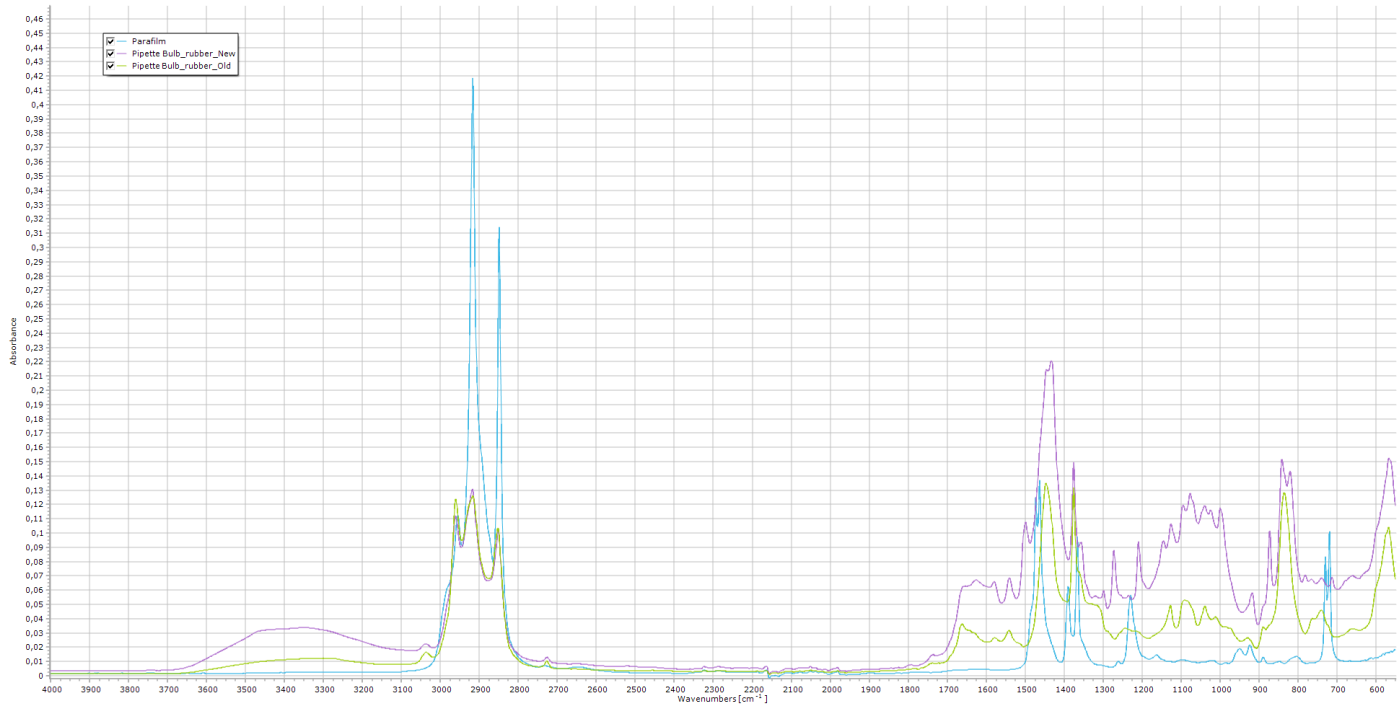

Figure 21. FTIR spectra of lab supplies; Parafilm, Old rubber pipette bulbs, and New rubber pipette bulbs. When pipetting Dichloromethane/Methanol, it is possible for the solution to react with the rubber and contaminate the sample. The lipid samples had been covered in parafilm for several months between extraction and filtering. Figure made with Spectragryph by Author.

If the samples contained a good amount of lipid, there should have been a peak around 3000cm^{-1}, as seen in FTIR done on Sperm Oil (see Fig. 22) and Raman spectra done on animal lipids (see Gao et al., 2021). Instead, they all show significant spikes around 1100-1000- and 600-500 cm^{-1}. The FTIR analysis of the lab supplies shows few similarities with the FTIR of the samples and mostly eliminates the possibility of them being the contamination source. When comparing these spikes to other FTIR studies done on sediments, such as Liu et al. (2013)(Fig. 23). It suggests that these samples contain mainly very fine-grained sediments and not lipids, which should be the contamination source and explain the age difference from the collagen sample.

As the lipid extraction step has been performed successfully previously at the lab and in other studies (Cequier-Sánchez et al., 2008), there is little reason to believe that the solvent failed to extract the lipid. More likely, there was little to no fat to extract to begin with. Before extracting the lipid from the sample, the bone is cleaned and ultrasonicated. It is thus also possible that the heat from the ultrasonication liquefies the fat, and therefore, the fat gets removed along with the superficial dirt.

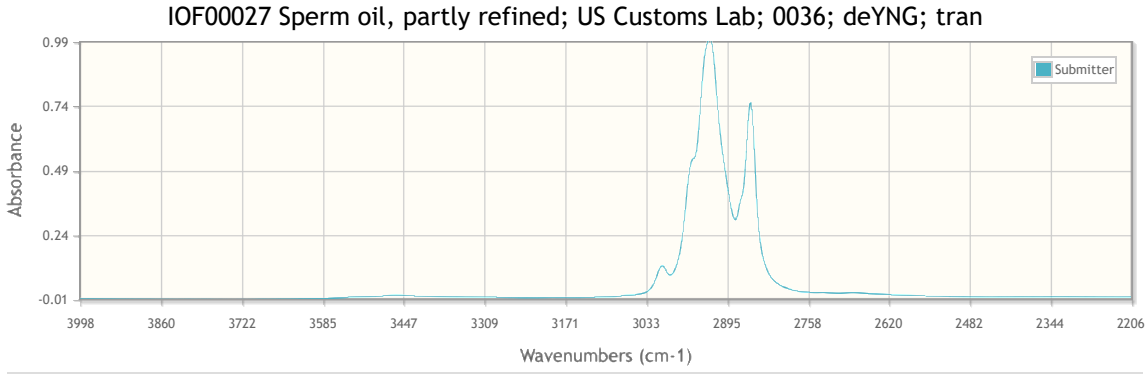

Figure 22. MFTIR (Fourier transform microspectroscopy) of Sperm Oil, peaks at 29920 cm^{-1}. (IRUG.org)

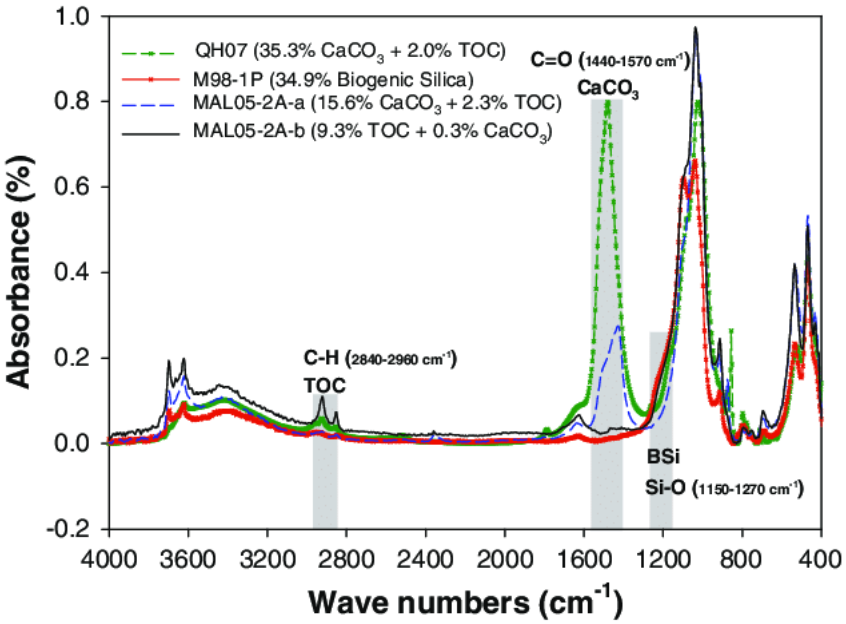

Figure 23. FTIR done on sediments. (Liu et al., 2013)

Chapter 6: Discussion

All the Bowhead Whale (*Balaena mysticetus*) samples (with delta corrections applied) date between 1587-1950 This places them in the commercial European whaling period in the Arctic region and until long after. As they are all from archaeological sites in the Netherlands, I will assume them to be products from Dutch whaling. However, I recognise that there is a possibility they could have been products from trade with indigenous people or other whaling nations such as England.[2]

[2] The Dutch already had a surpluss of whalebones from their own activities. I therefore see it as improbable they would try and acquire more through trade.

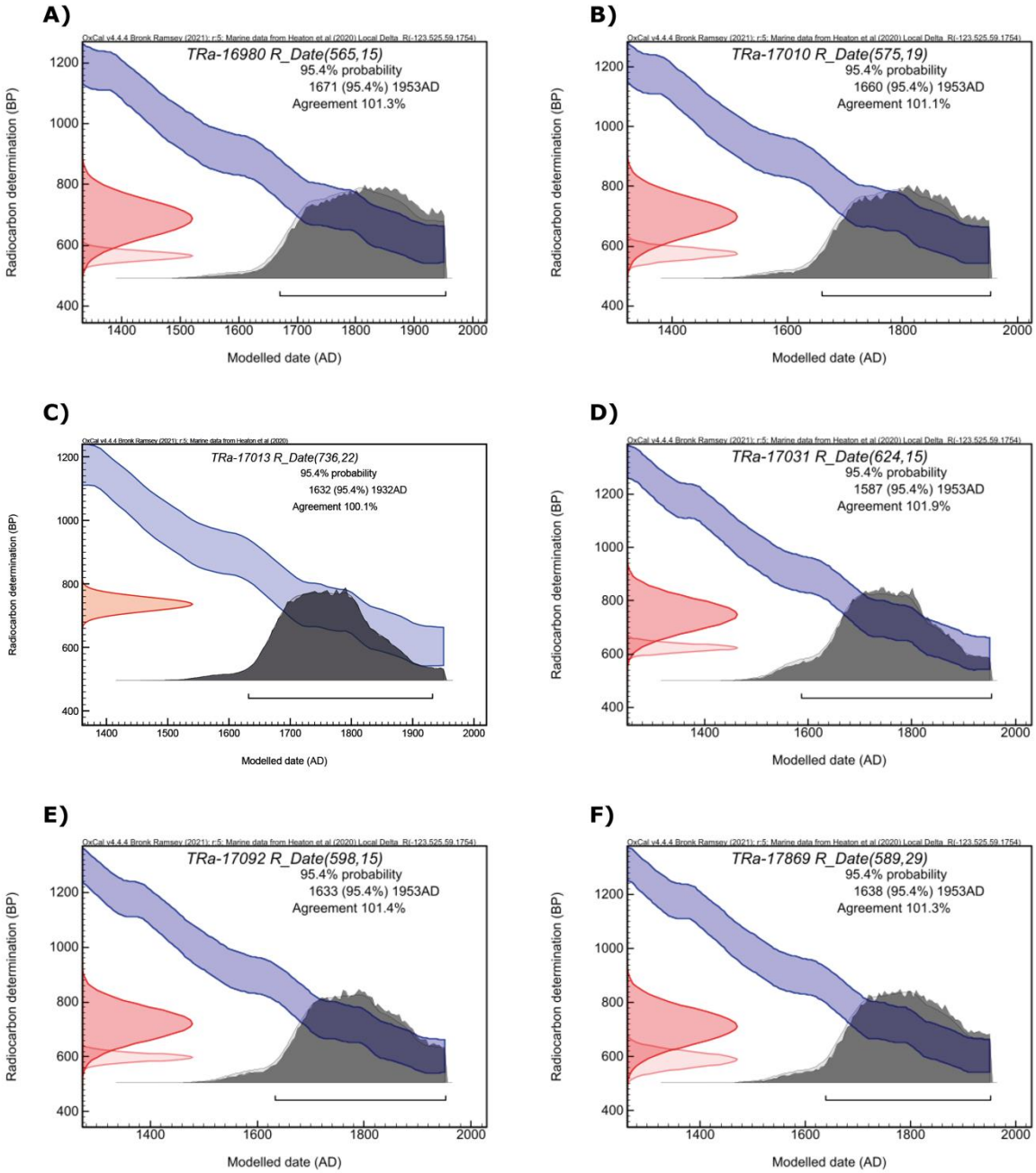

Figure 24. ΔR corrected Bowhead Samples individually calibrated in OxCal v.4.4.4 program.

6.1 TRa-16980, TRa-17010, TRa-17013, TRa-17031, TRa-17092, TRa-17869

As the Dutch did not begin whaling in the Arctic before 1612, it is unlikely that TRa-17031 is from before this. For all of the other samples', the earliest dates (the lower boundaries of the probability distributions in Fig.24), on the other hand, are plausible because the Dutch had already established themselves in the Arctic by that time. The results extend until the mid-20th century, which is beyond when Bowhead Whales were still hunted by the Dutch (Kruse, 2017). Dutch whaling had severely declined before the turn of the 19th century. When whaling resumed in the 19th century, it was largely unsuccessful, and one of the reasons for this is the lack of whales in their old hunting grounds. The Bowhead Whale (*Balaena mysticetus*) stock was still severely depleted. Of the few whales they caught, most of them are identified as Fin Whales (*Balaenoptera physalus*) and not Bowhead Whales. This is also supported by the catch data collected by Frigga Kruse, 2017 (Fig. 25). The catch data shows that the Bowhead Whale (*Balaena mysticetus*) was still caught between 1800-1850, but not by the Dutch. It is still possible for them to be from this time period as they might be from trade, but it is more likely that they stem from the Dutch whaling between 1612-1800. Therefore I would conclude that these bones are from the period 1612-1800 and are a direct result of the Dutch Whaling in the Arctic. This interpretation is entirely consistent with the radiocarbon dating evidence.

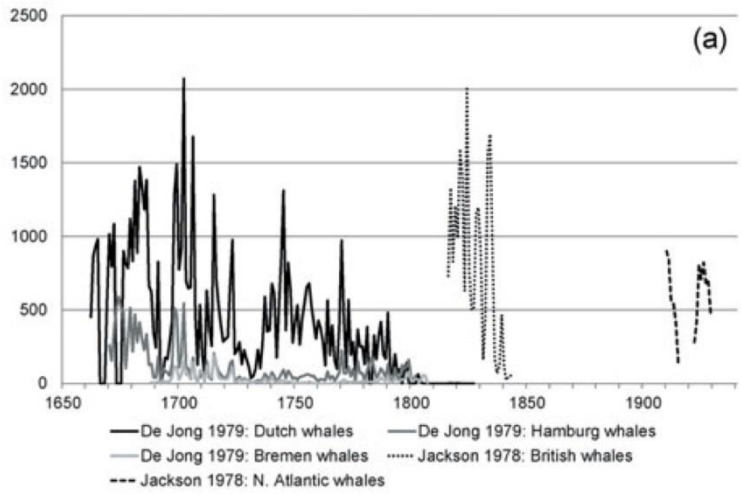

Figure 25. Line chart showing the catches per year of Bowhead Whale (Balaena mysticetus) from Kruse, 2017. The Dutch catch of the Bowhead Whale ends around 1800. (Kruse, 2017)

6.2 TRa-17013 -Smeerenburg

The TRa-17013 sample was collected at the archaeological site Smeerenburg during a survey and is assumed to be contemporary with the use of this site. Therefore TRa-17013 cannot be younger than 1619 or older than 1660. This made TRa-17013 appropriate for estimating possible ΔR values (see chapter 4.2.4 and 5.2).

6.3 The Importance of species-specific ΔR

As already noted in Chapter 1 (see 1.2.3), a species-specific ΔR is necessary to get an as accurate date as possible. If we look at the TRa-16980 sample (see Table 5), the youngest possible date given with only the use of the Marine20 curve was 1882 CE, which is after the period when the Dutch hunted Bowhead Whales (*Balaena mysticetus*). After applying the ΔR correction, the youngest possible date is 1671 CE which is much more likely with the context. Applying the typical ΔR correction for the Arctic of -400 based on marine molluscs could significantly alter the date of the whale bone and potentially shift its historical context, leading to a different interpretation altogether. This is also why I did not interpret the dates for the other species without a ΔR.

Sample ID	14C Age (rounded)	Calibrated Age Ranges	ΔR Corrected Age Ranges
TRa-16980	565 ± 15	68.3% probability 1882AD (68.3%) 1950AD 95.4% probability 1918AD (95.4%) 1950AD	95.4% probability 1671 (95.4%) 1953 AD
TRa-17010	575 ± 20	68.3% probability 1876AD (68.3%) 1950AD 95.4% probability 1919AD (95.4%) 1950AD	95.4% probability 1660 (95.4%) 1953 AD
TRa-17013	735 ± 20	68.3% probability 1676AD (68.3%) 1826AD 95.4% probability 1632AD (95.4%) 1935AD	95.4% probability 1619 (95.4%) 1659 AD
TRa-17031	625 ± 15	68.3% probability 1817AD (68.3%) 1950AD 95.4% probability 1869AD (95.4%) 1950AD	95.4% probability 1587 (95.4%) 1953 AD
TRa-17092	600 ± 15	68.3% probability 1854AD (68.3%) 1950AD 95.4% probability 1921AD (95.4%) 1950AD	95.4% probability 1633 (95.4%) 1953 AD
TRa-17869	590 ± 30	68.3% probability 1858AD (68.3%) 1950AD 95.4% probability 1918AD (95.4%) 1950AD	95.4% probability 1638 (95.4%) 1953 AD

Table 5. Table showing the Age ranges, both uncalibrated and calibrated with Marine20 for the samples with and without ΔR correction.

Chapter 7: Conclusion

This master thesis aimed to demonstrate the relevance of radiocarbon dating whale bones discovered in archaeological contexts and the significance of a species-specific ΔR. It also aimed to test the theory that the whale bones' protein and fat content would provide different ages due to the turnover rate as this is something that is rarely considered.

19 of the 20 collagen samples were successfully dated and calibrated using the Marine20 curve. One failed due to an insufficient amount of collagen. None of the Gray Whale (*Eschrichtius robustus*) or North Atlantic Whale (*Eubalena glacialis*) samples were from a known age context, so they will have to be corrected when a species-specific ΔR is available. As one of the Bowhead Samples was from a known age context, I was able to establish a species-specific ΔR for the Bowhead Whale (*Balaena mysticetus*). The ΔR correction is expressed as the interval (-225, -20) or as the ΔR value (-124 ± 59). With the ΔR correction applied to the other Bowhead Whale samples, it was possible to conclude that the bones most likely stem from the period 1612 - 1800 CE and are a direct result of the Dutch Whaling in the Arctic.

The uncorrected dates did not coincide with the possible historical contexts. And if one were to apply the typical ΔR correction for the Arctic of -400, based on marine molluscs, the date would get significantly altered and possibly be interpreted as a different historical context altogether. This demonstrated the importance of using a species-specific ΔR when dating marine mammals.

I did not, however, manage to test the theory that whale bones' protein and fat content would provide different ages. When the lipid samples came back with strange ^{14}C ages, it became apparent that something had gone awry in the process. FTIR- spectra of the remaining samples showed that the samples most likely contained little if any, lipids. Instead, they contained sediments. Therefore, it was not possible to compare the lipid dates with the collagen. It seems unlikely that the method was the source of contamination as similar methods had been used successfully before at the lab. Most likely, the sediments got radiocarbon dated instead of lipid, if there were any lipid at all.

It is unclear if this means that the method will not be usable on archaeological samples in the future. It is difficult to determine if the sediment type is a factor in the contamination since the samples were taken from different contexts. Despite being filtered multiple times, the sediment particles may be so fine that they still passed through the filters, making it difficult to prevent contamination through filtration

alone. It is also unclear what caused the low level of lipids in the bones. It either was very little or no lipid content in the bones. If the little lipid the bones contained got washed away with the water due to the low melting point of lipid, one might try a lower temperature. Not washing the bone at all before extraction would be problematic in other ways. But it is likely that there already was a lack of lipid from the start, and it is hard to say if that's because the lipid has disappeared due to time or context.

If the method is to be further tested, I would suggest making sure the bones have come from various contexts so that it is possible to figure out whether various sediments are contributing to sample contamination or if only certain types are responsible. It should also be made a point to collect from different time periods. This will assist in identifying if the problem is caused by poor preservation of the lipid or not.

This means that the thesis failed in its goal to see if the inbuilt age of the animal affects the dating. It did, however, succeed in dating 19 whale bones. Six of which I was able to put in a historical context after ΔR corrected the dates. This has given us valuable information regarding the circumstances surrounding the hunting of the whales the bones stem from. This includes details on the location of the hunt, the motives behind it, and the individuals or groups responsible for carrying it out. When ΔR values are determined for the other species and the dates are updated, the same information may be obtained from these as well.

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Appendix 1 Collagen Protocol

Bone sample pre-treatment – Youri van den Hurk

Bone procedure:

DAY 1

1. Visual inspection of the sample to remove carbon contaminants (roots, textile fibers, hair
etc.) and inspect whether the sample has been consolidated with glue

1b. Bone that have been consolidated with glue needs to soaked in water at room temperature with periodic mixing. After 24 hours they need to rinsed thoroughly with water and dried under vacuum. Soaking solutions and rinse water need to be pooled and freeze-dried to recover the soluble materials.

2. Crushing of sample to small pieces. Start with 0.5 gram of sample for collagen preparation. Leave 0.1-0.2 gram for aDNA analysis if possible in the original plastic bag with the specimen NTNU label on it. Weigh a tube and put the label and the scotch tape to hold the label on the tube. Weigh the tube (with the label and tape). Put the sample in it and weigh again.

3. Clean the bone piece with water (3x5 min, or until clean) in the ultrasonic bath. Let the
bone dry (not in the oven!) After evaporation of all water, weigh the tube with the sample again.

DAY 2

~~4. Washing with acetone (30 min). Put aluminium foil, cover the tubes and make a small hole~~
~~in the film, put in desiccator and pump slowly to accelerate drying.~~

4b. Lipid has to be removed by dichloromethane and methanol (2:1). Wear thick gloves and work in the fume hood. Submerge your sample in the mixture and sonicate for 15 mins, making sure to cool the bath with ice since DCM will boil at about 40dC. Repeat the process at least 3 time up until you no longer have colour in the solution, changing solution each time. Let the sample dry overnight before moving on to step 5.

4c. Weigh another tube (with the lipid label and the scotch tape). Collect the lipid in small vail. Let the dichloromethane and methanol evaporate for a couple of days. Weigh again after all the lipid has evaporated.

DAY 3

5. Demineralization with 1.125 ml “Youri bone concentration” HCL (10 ml HCL (conc.) in 40 ml water) per 100 mg of bone. For smaller specimens (up to 300 mg) use normal “Bone concentration” HCL (10 ml HCL (conc.) in 90 ml water) per 100 mg of bone.
6. Put Parafilm cover on the tubes and make a small hole in the Parafilm
7. Put in desiccator and pump slowly to avoid foaming to the top of the tubes.

DAY 4

8. Pump every 15 minutes to accelerate demineralization. Check pH after about 2 h, or when
it stops bubbling, and add 2-3 drops of concentrated acid if pH>1. Make sure the bone
pieces stuck on the walls are pushed back into the acid.
9. After bubbling has stopped, wait 30 min and check again. If bubbling continues, go back to
step 8 until it totally stops. The sample should be in light flakes. If the sample contains
hard pieces, add 2-3 drops of concentrated acid and pump again until bubbling stops.
10. Remove acid with pipette but leave some liquid above the sample to avoid pipetting the
sample (1 mm). HCL goes in the Saltsyre bottle.
11. Wash with distilled water (4-5 times) and wait 30 minutes with the sample in water.
Measure pH, if pH=3-4 continue to step 12. If pH<3, wash again and wait for 30 minutes
before measuring the pH again.
12. Remove the extra water with a pipette. Leave some liquid above the sample. If you leave
the sample for the night, leave slightly more water on the top of it.
13. Add 4 ml NaOH (0.125 M; 0.5 %) at room temperature for **no more than 4 h** (2 h usually
depending on the colour of the sample).
14. Remove NaOH solution with a pipette. Leave some liquid above the sample. Humic acid
(dissolved fraction **A**) can be separated at this point if the solution looks brownish, see
further pretreatment for this fraction below.

15. Wash with distilled water (3-4 times) and wait 30 minutes. If pH<10 proceed to step 16, if
not wash again. When leaving for weekend, 2 drops of “bone concentration” in tube.

DAY 5

16. Add 5 ml “bone concentration” HCL at room temperature for 1-2 hours to remove
atmospheric CO_2. The final pH should be 1.
17. Wash with distilled water to achieve pH 3.0±0.2 in the solution. If larger than 3, you can
add a drop of the “bone concentration” HCL to bring the pH down if too much water was
added. The sample should be in 5 mL at that point.
18. Put the tube with the sample at pH 3 in the oven at 70 °C overnight to hydrolyze the
collagen to gelatin. The solution should be transparent when the collagen is hydrolyzed.
There might be small particles left at the bottom.

DAY 6

19. Filter the gelatin using a quartz aerosol filter (99.998 % capture for 0.3 µm particles) at
70 °C. Bake the filter before use.
20. Filtrate. The filter with the residue is dried in the oven at 70 °C over night. Keep the filter and the residue in aluminium foil in a plastic bag for potential further analysis.
21. Weight the collagen and put in small flask

DAY 7

21. The filtrated gelatin is frozen first in the freezer in a tilted position, and then freeze-dried.
This fraction should be called **collagen**. Remove the collagen and put an aluminium foil to weigh the collagen. Then put collagen in small flask with the collagen label and weight of the collagen on it.

Procedure for fraction A:

1. Precipitate dissolved fraction by adding 4.4 M (37 %) HCl until the solution is clear (pH<1).
2. Rinse the precipitate with distilled water until pH>3 (≈3 times).

3. Dry the sample in the oven at 70 °C. This fraction should be called **humic acids**. Discard the humic acids.

Acid calculation:

$CaCO_3 + 2HCl > CaCl_2 + H_2CO_3 > CaCl_2 + CO_2 + H_2O$

Removing the CO_2 prevents the reaction to go backwards and produce $CaCO_3$ again.

$CaCO_3$ in 100 mg per milli-mole

Concentrated HCl (37%) = 10 milli-mole per ml.

We need 2 milli-mole concentrated HCl for 100 mg bone

So we need 0.2 ml concentrated HCl.

To avoid the strong reaction with concentrated acid and the lack of liquid, we dilute

the acid (1/10 or 10 ml concentrated in 90 ml water) = "bone concentration"

So we need 2.0 ml (0.2 ml x 10) of "bone concentration" per 100 mg bone.

As we need to have extra H^+ ions, **we use 2.5 ml per 100 mg bone**.

Glue Samples

For samples with BISON woodglue (Glue based on PVAC – 12 specimens of which 3 need to get dated and all need stable isotope analysis)

- Rinse 3 times with hot water (40-50 degrees) on hot plate

For samples with velpon with acetone (velpon is neoprene rubber based glue – treated with just a thin layer (not impregnated) – all for stable isotope analysis)

- Sample not near surface
  - Petroleum ether – 3 rep → 80 degrees
  - Acetone 10 min – 3 rep. → 90 degrees
  - Methanol 10 min – 3 rep → 90 degrees
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Appendix 2 Lipid Protocol

Lipid Pre-treatment

Step 1

1) Pre weigh one tube and one filter per sample.
   - The filters need to be baked the day before for 6 hours to sterilize

2) Filter the lipid sample.
2a) First liquify the lipid by adding Dichloromethane/Methanol (2:1). Amount of solution does not matter as long as the sample is covered. Make sure to also rinse the sides. Sonicate for 15 minutes. Make sure to cool down the water beforehand as Dichloromethane/Methanol boils at around 39˚C.
2b) Filter the lipid using (Merck Millipore, AQFA04700, 99.998 % for 0.3 µm particles). Pipette the lipid slowly to avoid any getting stuck in the funnel. Rinse the funnel with Dichloromethane/methanol after removing the filter.
3) The filter with the lipid should be left to dry in the oven at 70˚C over night. The tubes should be left to dry in the fume hood.

Step 2

4) Weigh the dried filters.
5) Prepare the “teabags”. The dried filters are folded into a small package by using sterilized tweezers and then attached to a sterilized paperclip. Afterwards transfer the teabags into the corresponding tube used during the first filtering.
6) “Mini Soxhlet”. (Maximum 5 samples at a time)
6a) The tubes need to be filled with Dichloromethane/methanol until the filter is completely covered. Then the tube needs to be closed with a silicone plug.
6b) Put the tubes into the “Mini Soxhlet” and set the temperature to 41˚C and leave for about one hour. Make sure to check in a few times to make sure the plugs stay in place.
7) Rinse thoroughly with Dichloromethane/Methanol when removing the “teabags” from the tubes.
8) Add new pre-baked and pre-weighed filter into the tube to absorb the Dichloromethane/Methanol

Leave to dry until the Dichloromethane/methanol has completely evaporated. (Approximately a week)

Step 3

10) Weigh filters to see if there is enough lipid for further dating, the weight should be >1 mg
11) Place filters in a pre-baked (850 °C for 6 hours) quartz tube (outer diameter: 9 mm, inner diameter: 7 mm, length: 180 mm. Multi-Lab Ltd, Newcastle upon Tyne, England) filled with 500 mg CuO pellets and a piece of silver (ca 100 mg Ag).
12) Follow NLAD protocol for Combustion and Cracking

Appendix 3 Combustion and Cracking Protocol

Sealing of combustion tubes

Date: 15.02.2016

Preparation:

1. A quartz tube with a diameter of 9 mm is used for combustion. The usual quartz tubes have the following dimensions: outer diameter: 9 mm, inner diameter: 7 mm, length: 180 mm. They are ordered from Multi-Lab Ltd (Newcastle upon Tyne, England).
2. The tube is filled with 500 mg CuO pellets.
3. Add a silver piece to it (about 100 mg Ag).
4. Bake the tube at 850 °C in the oven for 6 hours.

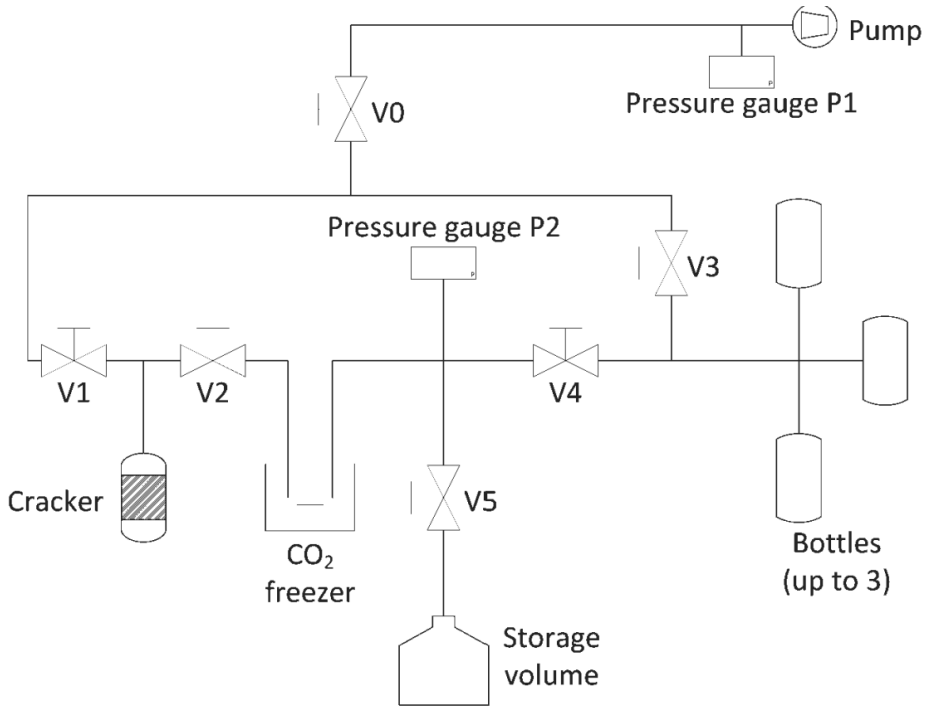
Sample sealing:

1. Fill the weighted amount of sample into an inset tube (diameter 6 mm).
2. Insert the sample tube into a pre-baked sealing tube (see preparation).
3. Add baked (850 °C, 4 hours) quartz wool to the tube if the sample material might be pumped away, e.g. is a fine powder.
4. Label the tube with the SID and CO2ID using the gold pen.
5. Mount the filled tube at the sealing line and start pumping.
6. Wait until the pressure is below $5 \cdot 10^{-5}$ mbar.
7. Close the valve to the tube to avoid venting the system when sealing results in a leak.
8. Seal the tube using the propane-oxygen flame.

Combustion:

The sealed combustion tubes are placed in the ceramic (Sillimantin 60) tubes inside the oven. The oven is ramped to 850 °C where it stays for 4 hours.

Tube cracking
Date: 28.09.2015
Updated: 29.06.2016



Cracker preparation:

Cracker layout:

Cracker preparation:

1. Mount combustion tube and bottles at the system.
2. Open all valves (including bottles) for pumping.
3. Wait until low pressure P1 is in low 10^{-5} mbar range.

Sample cracking (no gas outtake for δ13C measurement):

1. Check pressure P2 for offset.
2. Close valves V1-V5.
3. Crack the combustion tube. Freeze water in combustion tube with dry ice slush.
4. Wait until the water is frozen out (2-5 minutes).
5. Open V2 and freeze CO_2 in the freezer loop with liquid nitrogen.
6. Wait until the pressure P2 returned to the offset value and the liquid nitrogen stops boiling (1-2 minutes).
7. Open V1 to pump non-condensed gases.
8. Wait until pressure P1 reaches low 10^{-5} mbar range.
9. Close V2 to separate CO_2 from the water.
10. Close V1 to isolate the cracker.
11. Remove liquid nitrogen and dry ice slush.
12. Open V3 and gas bottle for pumping.

13. Wait until CO2 has evaporated completely (pressure P2 stable). Applying hot water to the freezer loop can accelerate the process.
14. Read pressure P2 to quantify the gas amount.
15. Apply dry ice slush to freezer loop to freeze water.
16. Wait until the water is frozen (1-2 minutes).
17. Check pressure P1 to be in low 10^{-5} mbar range to ensure empty bottle.
18. Close V3.
19. Open V4 and freeze CO_2 in bottle with liquid nitrogen.
20. Wait until pressure P2 is down to the offset value and liquid nitrogen is not boiling.
21. Open V3 to pump air from bottle.
22. Wait until pressure P1 is low enough.
23. Close gas bottle.
24. Remove liquid nitrogen from bottle.
25. Close V3 and V4.
26. Remove gas bottle from system.

Sample cracking (with gas outtake for δ13C measurement):

1. Check pressure P2 for offset.
2. Close valves V1-V5.
3. Crack the combustion tube. Freeze water in combustion tube with dry ice slush.
4. Wait until the water is frozen out (2-5 minutes).
5. Open V2 and freeze CO_2 in the freezer loop with liquid nitrogen.
6. Wait until the pressure P2 returned to the offset value and the liquid nitrogen stops boiling (1-2 minutes).
7. Open V1 to pump non-condensed gases.
8. Wait until pressure P1 reaches low 10^{-5} mbar range.
9. Close V2 to separate CO_2 from the water.
10. Close V1 to isolate the cracker.
11. Remove liquid nitrogen and dry ice slush.
12. Open V3 and gas bottle for pumping.
13. Wait until CO2 has evaporated completely (pressure P2 stable). Applying hot water to the freezer loop can accelerate the process.
14. Open V5 to expand gas in storage volume.
15. Close V5 to separate gas for d13C analysis.
16. Read pressure P2 to quantify the gas amount (for AMS measurement).
17. Apply dry ice slush to freezer loop to freeze water.
18. Wait until the water is frozen (1-2 minutes).
19. Check pressure P1 to be in low 10^{-5} mbar range to ensure empty bottle.
20. Close V3.
21. Open V4 and freeze CO_2 in bottle with liquid nitrogen.
22. Wait until pressure P1 is down to the offset value and liquid nitrogen is not boiling.
23. Open V3 to pump air from bottle.
24. Close gas bottle.
25. Remove liquid nitrogen from bottle.
26. Close V4.
27. Open V5 and apply liquid nitrogen to freezer loop.

28. Close V5 when CO_2 is frozen.
29. Evaporate CO_2 to measure pressure P2 for $\delta^{13}C$ measurement.
30. Open V4 and bottle. Freeze CO_2 to bottle with liquid nitrogen.
31. Close V3 and V4.
32. Remove gas bottles from system.

Appendix 4 OxCal Code DeltaR

```
Options()
{
 Curve="Marine20.14c";
};
Plot()
{
 R_Date("TRa-17013",736,22);
 Sequence("Bowhead Whale 1")
 {
  Delta_R("Local1",U(-225,-20));
  Boundary("Start 1");
  C_Date("start hunting 1",1619,1);
  R_Date("TRa-17013 DeltaR = ",736,22);
  C_Date("stop hunting 1",1660,1);
  Boundary("End 1");
 };
 Curve("Marine20.14c");
 Delta_R("Local",U(-225,-20));
 R_Date("TRa-16980",565,15);
 R_Date("TRa-17010",575,19);
 R_Date("TRa-17031",624,15);
 R_Date("TRa-17092",598,15);
 R_Date("TRa-17869",589,29);
 R_Date("TRa-17013",736,22);
};
```

Appendix 5 Collagen Results from NLAD

National Laboratory for Age Determination
14C Result Report

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Measurement references:
Seiler et al., Radiocarbon 61(6), 2019

Calibration references:
OxCal v4.4.2 Bronk Ramsey (2020); r:5
Marine data from Heaton et al (2020)

Sample Name	Fraction	14C content (pMC)	14C Age (rounded)	d13C (from AMS system)	Calibrated Age Ranges	mgC	Fraction Yield(%)	C content % by weight	N Content % by weight	C:N ratio by weight	% Current	14C Age (not rounded)
TRa-16922 DAG191	Collagen	56.42 ± 0.27	4600 ± 40	-14.9 ± 0.8 ‰	68.3% probability 2772BC (68.3%) 2569BC 95.4% probability 2857BC (95.4%) 2488BC	0.51	1	17	6,2	2,8	66,2	4598 +42/-41 BP
TRa-16922 DAG191	Collagen	56.12 ± 0.20	4640 ± 35	-15.4 ± 0.8 ‰	68.3% probability 2832BC (68.3%) 2647BC 95.4% probability 2886BC (95.4%) 2553BC	0.44	1	17	6,1	2,8	48,1	4641 +35/-35 BP
TRa-16922 DAG191	*Average:*	*56.27 ± 0.24*	*4620 ± 35*	*-15.2 ± 0.8*	*68.3% probability 2811BC (68.3%) 2620BC 95.4% probability 2872BC (95.4%) 2542BC X2-Test: df=1 T=0.6(5% 3.8)*							
TRa-16925 DAG195	soil, porous, Collagen	58.53 ± 0.13	4305 ± 20	-14.7 ± 1.2 ‰	68.3% probability 2372BC (68.3%) 2198BC 95.4% probability 2451BC (95.4%) 2129BC	1.30	13	45	17	2,7	96,5	4303 +20/-20 BP
TRa-16980 DAG251	bone, Collagen	93.21 ± 0.17	565 ± 15	-13.2 ± 1.4 ‰	68.3% probability 1882AD (68.3%) 1950AD 95.4% probability 1918AD (95.4%) 1950AD	1.41	11	46	17	2,7	94,4	565 +15/-15 BP
TRa-16988 DAG335	Bone and soil, Collagen	63.81 ± 0.13	3610 ± 20	-15.2 ± 1.4 ‰	68.3% probability 1464BC (68.3%) 1321BC 95.4% probability 1525BC (95.4%) 1249BC	1.22	4	39	14	2,9	111,3	3609 +18/-18 BP
TRa-16990 DAG342	Bone and a lot of soil, Collagen	86.38 ± 0.14	1175 ± 15	-13.3 ± 1.3 ‰	68.3% probability 1312AD (68.3%) 1413AD 95.4% probability 1270AD (95.4%) 1464AD	1.42	2	42	15	2,7	116,7	1176 +14/-14 BP
TRa-16999 DAG46	Bone and soil, Collagen	81.07 ± 0.18	1685 ± 20	-15.8 ± 1.4 ‰	68.3% probability 796AD (68.3%) 942AD 95.4% probability 726AD (95.4%) 1006AD	1.58	5	47	17	2,7	111,6	1686 +19/-18 BP
TRa-17000 DAG50	Bone and soil, Collagen	82.76 ± 0.15	1520 ± 15	-15.7 ± 1.3 ‰	68.3% probability 985AD (68.3%) 1121AD 95.4% probability 907AD (95.4%) 1180AD	1.38	4	45	16	2,7	107,5	1520 +15/-15 BP
TRa-17002 DAG160	Bone and soil, Collagen	91.94 ± 0.22	675 ± 20	-12.1 ± 1.9 ‰	68.3% probability 1734AD (68.3%) 1898AD 95.4% probability 1696AD (95.4%) 1950AD	0.92	15	33	12	2,7	66,1	675 +20/-20 BP
TRa-17010 DAG247	Bone and soil, Collagen	93.09 ± 0.21	575 ± 20	-15.5 ± 1.2 ‰	68.3% probability 1876AD (68.3%) 1950AD 95.4% probability 1919AD (95.4%) 1950AD	1.33	4	44	16	2,7	98	575 +19/-19 BP
TRa-17013 DAG298	Dense bone, Collagen	91.25 ± 0.25	735 ± 20	-14.5 ± 1.4 ‰	68.3% probability 1676AD (68.3%) 1826AD 95.4% probability 1632AD (95.4%) 1935AD	1.22	8	44	16	2,7	93,8	736 +22/-22 BP
TRa-17027 DAG54	Bone and soil, Collagen	79.92 ± 0.15	1800 ± 15	-13.9 ± 1.4 ‰	68.3% probability 681AD (68.3%) 805AD 95.4% probability 646AD (95.4%) 887AD	1.20	8	40	15	2,7	98,2	1801 +17/-16 BP
TRa-17031 DAG96	Bone and soil, Collagen	92.53 ± 0.16	625 ± 15	-15.1 ± 1.4 ‰	68.3% probability 1817AD (68.3%) 1950AD 95.4% probability 1869AD (95.4%) 1950AD	1.49	4	45	16	2,8	109,5	624 +15/-15 BP
TRa-17092 DAG75	Bone and soil, Collagen	92.83 ± 0.17	600 ± 15	-13.4 ± 1.3 ‰	68.3% probability 1854AD (68.3%) 1950AD 95.4% probability 1921AD (95.4%) 1950AD	1.19	5	40	15	2,7	94,1	598 +15/-15 BP
TRa-17134 DAG92	Bone and a lot of soil, Collagen	77.96 ± 0.15	2000 ± 15	-14.9 ± 1.3 ‰	68.3% probability 490AD (68.3%) 623AD 95.4% probability 430AD (95.4%) 669AD	1.19	4	38	14	2,7	99,2	2000 +17/-17 BP
TRa-17814 DAG742	Bone pieces and powder, Collagen	83.91 ± 0.16	1410 ± 15	-14.7 ± 1.3 ‰	68.3% probability 1091AD (68.3%) 1225AD 95.4% probability 1039AD (95.4%) 1277AD	1.16	6	35	13	2,8	97,6	1409 +16/-16 BP
TRa-17847 DAG439	Bone and soil, Collagen	91.39 ± 0.16	725 ± 15	-14.5 ± 1.2 ‰	68.3% probability 1687AD (68.3%) 1836AD 95.4% probability 1650AD (95.4%) 1932AD	1.56	3	45	17	2,6	108,1	723 +15/-15 BP
TRa-17869 DAG463	Bone, Collagen	92.93 ± 0.33	590 ± 30	-14.6 ± 1.4 ‰	68.3% probability 1858AD (68.3%) 1950AD 95.4% probability 1918AD (95.4%) 1950AD	0.99	7	31	11	2,8	88	589 +29/-29 BP
TRa-18015 DAG534	Collagen	91.23 ± 0.15	735 ± 15	-15.5 ± 1.5 ‰	68.3% probability 1679AD (68.3%) 1821AD 95.4% probability 1631AD (95.4%) 1927AD	1.36		43	16	2,7	116,1	737 +14/-14 BP
TRa-18032 DAG551	Collagen	90.75 ± 0.20	780 ± 20	-15.9 ± 1.1 ‰	68.3% probability 1647AD (68.3%) 1799AD 95.4% probability 1550AD (95.4%) 1866AD	0.68		40	14	2,9	65,8	780 +20/-20 BP

Appendix 6 Lipid Results

National Laboratory for Age Determination
14C Result Report

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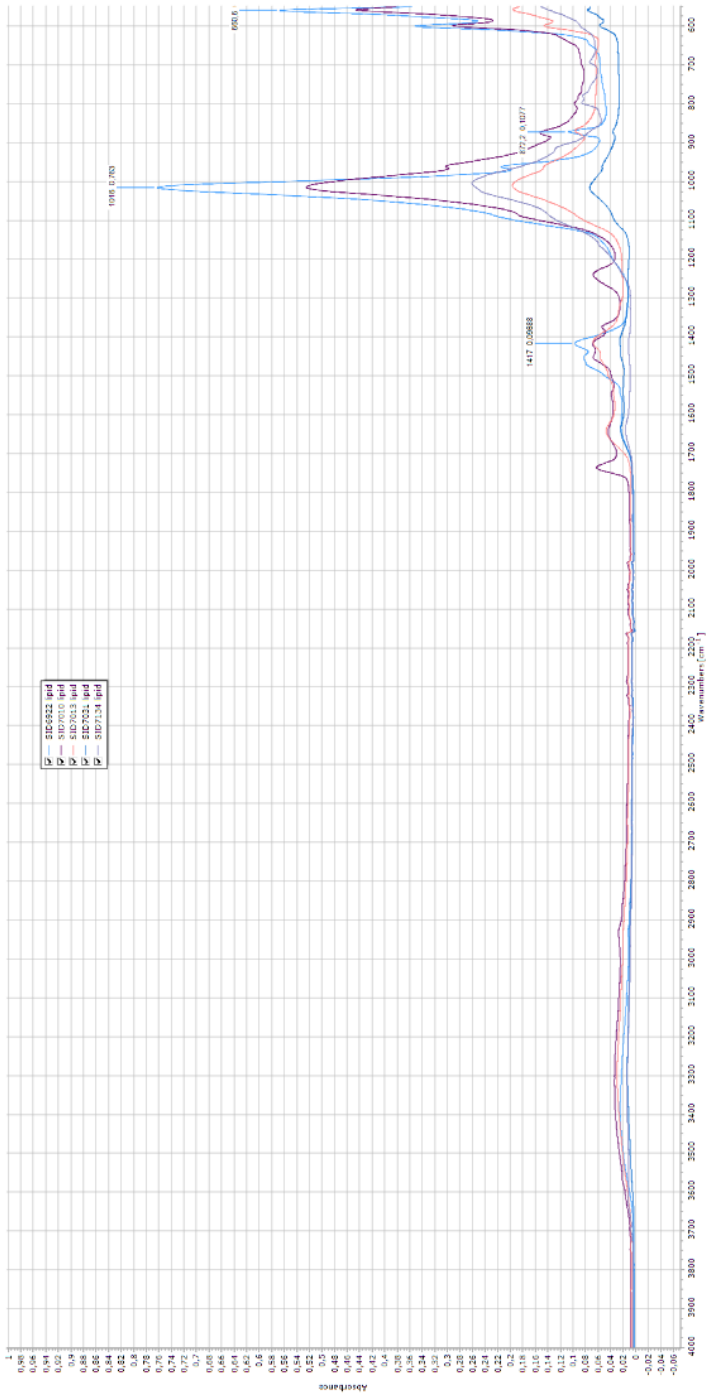
Measurement references:
Seller et al., Radiocarbon 61(6), 2019

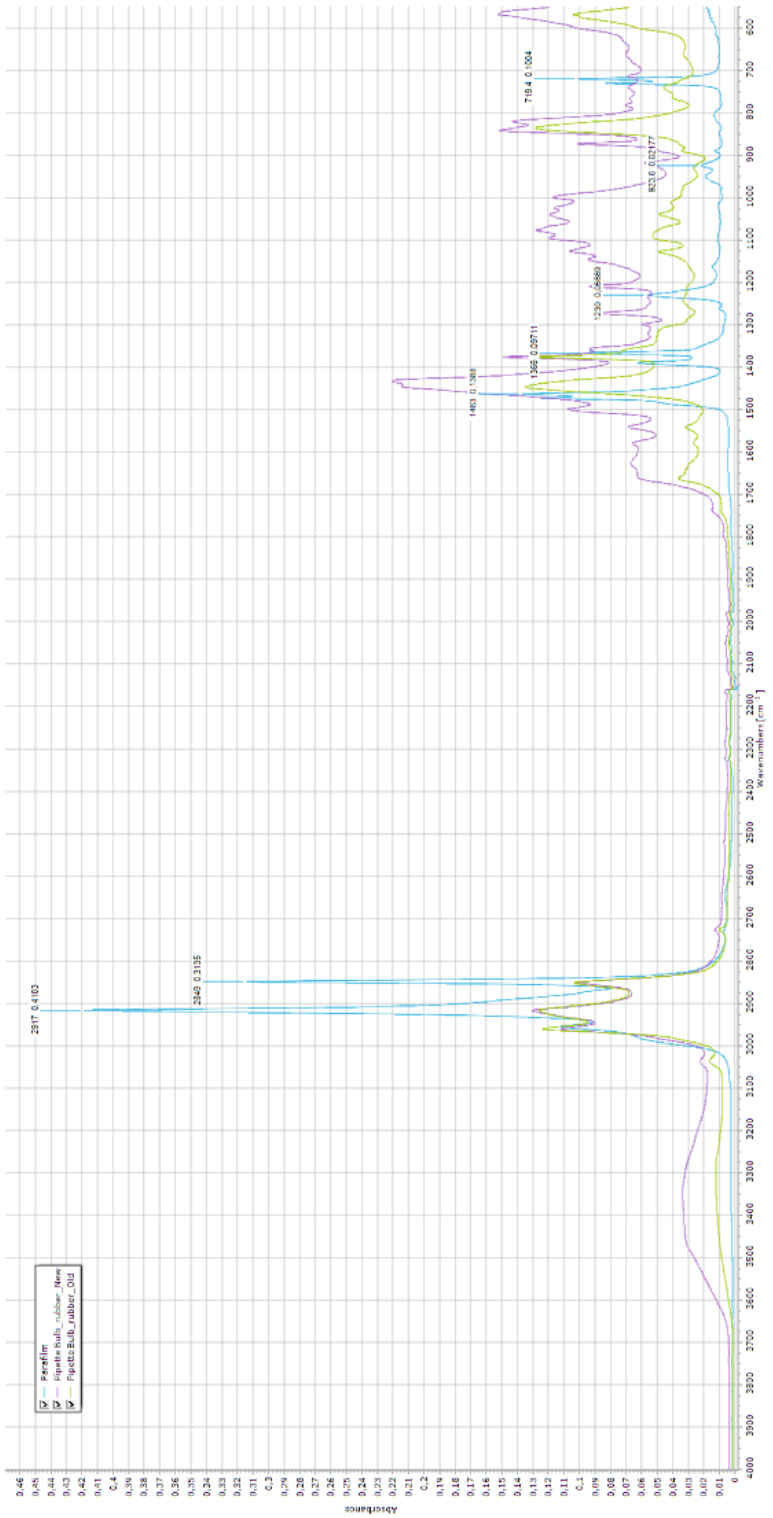
Calibration references:
OxCal v4.4.2 Bronk Ramsey (2020); r:5
Marine data from Heaton et al (2020)

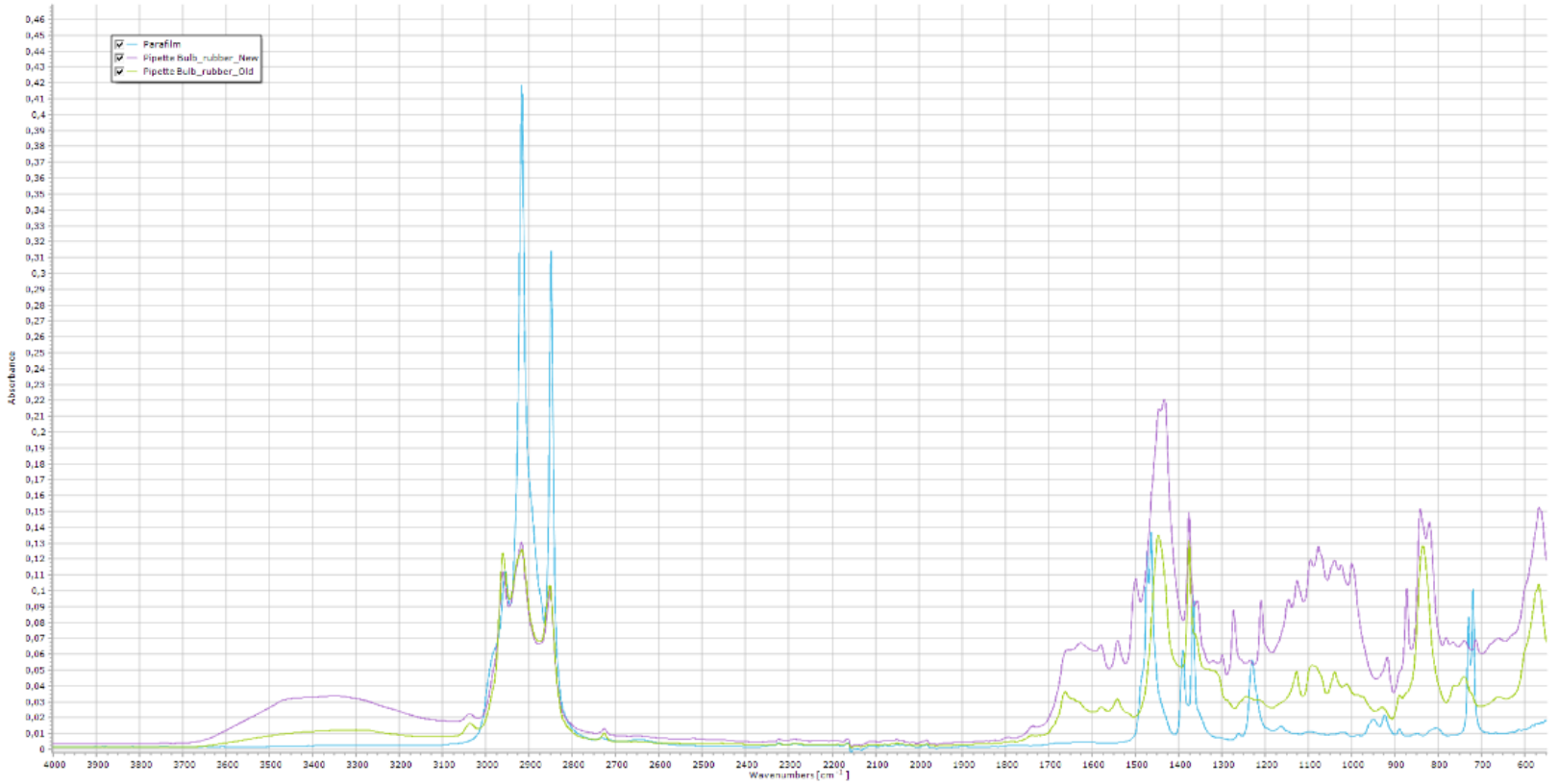
Sample Name	Fraction	14C content (pMC)	14C Age (rounded)	d13C (from AMS system)	Calibrated Age Ranges	% C	mgC	Yield (%)	Fraction by weight	C content % by weight	N Content % by weight	C/N ratio by weight	% Current	14C Age (not rounded)	Run date
					2008AD (68.3%) 2013AD										
					95.4% probability										
					1952AD (9.8%) 1958AD										
TRo-20706 Whale bone 1	Bone, Collagen	104.82 ± 0.14	> 1950 AD	-17.1 ± 0.1 ‰	2001AD (85.7%) 2015AD	46	1.30		47	17	3.7	59.6	> 1950 AD	2023-04-24	
					68.3% probability										
					2008AD (68.3%) 2013AD										
					95.4% probability										
					1952AD (16.5%) 1958AD										
TRo-20706 Whale bone 1	Bone, Lipid	104.01 ± 0.15	> 1950 AD	-26.0 ± 3.7 ‰	2001AD (83.0%) 2015AD	57	1.98		57	0.04	1532.1	140.6	> 1950 AD	2023-05-02	
					68.3% probability										
					2008AD (68.2%) 2013AD										
					95.4% probability										
					1952AD (11.2%) 1958AD										
					2004AD (87.2%) 2015AD										
TRo-20706 Whale bone 1	Average:	104.42 ± 0.15	> 1950 AD	-21.6 ± 1.9 ‰	X2-Test: df=1 T=15.65% 3.8]										
					68.3% probability										
					2008AD (68.3%) 2013AD										
					95.4% probability										
TRo-20707 Whale bone 2	Bone, Collagen	104.93 ± 0.13	> 1950 AD	-16.6 ± 0.2 ‰	1952AD (10.1%) 1959AD	46	1.41		46	17	2.8	107.7	> 1950 AD	2023-04-24	
					2009AD (84.0%) 2015AD										
					68.3% probability										
					2008AD (68.2%) 2013AD										
					95.4% probability										
					1953AD (11.2%) 1958AD										
TRo-20707 Whale bone 2	Bone, Lipid	104.25 ± 0.18	> 1950 AD	-27.0 ± 0.3 ‰	2006AD (84.2%) 2015AD	79	1.58		79	0.08	1046.3	105.4	> 1950 AD	2023-05-06	
					68.3% probability										
					2008AD (68.3%) 2013AD										
					95.4% probability										
					1953AD (8.1%) 1958AD										
TRo-20707 Whale bone 2	Average:	104.59 ± 0.16	> 1950 AD	-22.8 ± 0.3 ‰	2007AD (87.3%) 2015AD										
					X2-Test: df=1 T=6.4033 3.8]										
					68.3% probability										
					2008AD (68.3%) 2013AD										
					95.4% probability										
					1953AD (8.1%) 1958AD										
TRo-20708 Whale bone 3	Bone, Collagen	104.67 ± 0.13	> 1950 AD	-16.4 ± 0.4 ‰	2006AD (87.3%) 2015AD	44	1.28		44	16	2.8	98.4	> 1950 AD	2023-04-24	
					68.3% probability										
					2008AD (68.3%) 2013AD										
					95.4% probability										
					1953AD (8.1%) 1958AD										
TRo-20708 Whale bone 3	Bone, Lipid	103.67 ± 0.16	> 1950 AD	-26.3 ± 2.2 ‰	2006AD (66.0%) 2017AD	44	1.32		44	0.03	1332.2	116.4	> 1950 AD	2023-05-02	
					68.3% probability										
					2008AD (68.2%) 2013AD										
					95.4% probability										
					1953AD (10.8%) 1958AD										
TRo-20708 Whale bone 3	Average:	104.17 ± 0.15	> 1950 AD	-21.4 ± 1.9 ‰	X2-Test: df=1 T=21.65% 3.8]										
					68.3% probability										
					2008AD (68.3%) 2013AD										
					95.4% probability										
TRo-20709 Whale bone 4	Bone, Collagen	104.69 ± 0.17	> 1950 AD	-16.9 ± 1.0 ‰	1953AD (8.8%) 1958AD	46	1.47		46	16	2.8	93.9	> 1950 AD	2023-04-24	
					2009AD (86.6%) 2015AD										
					68.3% probability										
					2008AD (68.2%) 2013AD										
					95.4% probability										
					1952AD (12.1%) 1959AD										
TRo-20709 Whale bone 4	Bone, Lipid	103.98 ± 0.15	> 1950 AD	-26.7 ± 3.7 ‰	2006AD (84.3%) 2015AD	47	1.69		47	0.03	1572.3	129.3	> 1950 AD	2023-05-02	
					68.3% probability										
					2008AD (68.2%) 2013AD										
					95.4% probability										
					1953AD (11.3%) 1958AD										
TRo-20709 Whale bone 4	Average:	104.32 ± 0.16	> 1950 AD	-21.8 ± 2.0 ‰	2004AD (84.4%) 2015AD										
					X2-Test: df=1 T=13.45% 3.8]										
					68.3% probability										
					2008AD (68.2%) 2013AD										
					95.4% probability										
					1952AD (10.2%) 1958AD										
TRo-20710 Whale bone 5	Bone, Collagen	104.94 ± 0.12	> 1950 AD	-14.8 ± 0.4 ‰	2001AD (85.2%) 2015AD	44	1.41		44	16	2.8	109.4	> 1950 AD	2023-04-24	
					68.3% probability										
					2008AD (68.2%) 2013AD										
					95.4% probability										
					1953AD (10.2%) 1957AD										
TRo-20711 Whale fat	Whale fat	105.74 ± 0.14	> 1950 AD	-19.3 ± 3.7 ‰	2001AD (85.3%) 2014AD	25	0.79		25	8.5	2.9	85.1	> 1950 AD	2023-04-24	

Appendix 7 FTIR

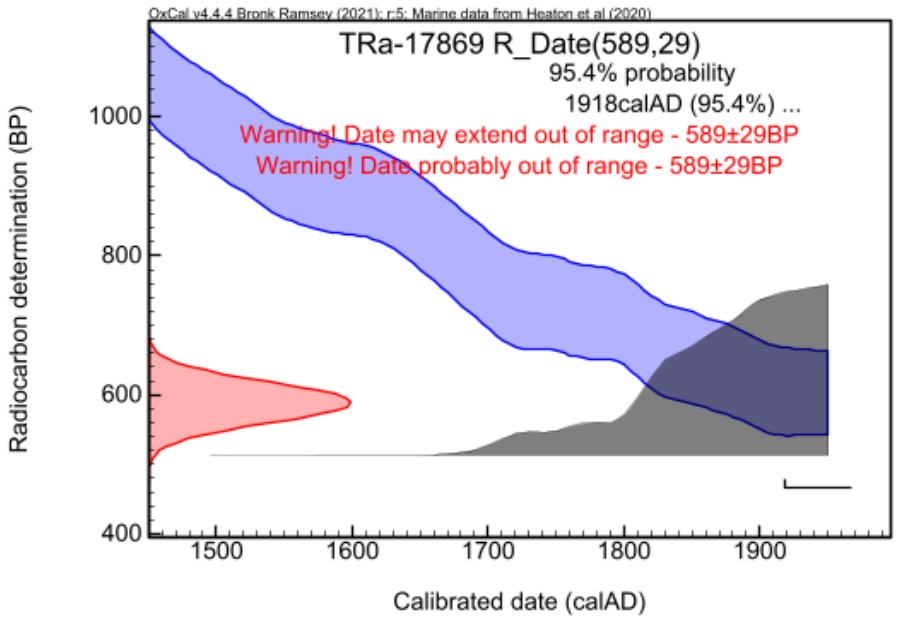


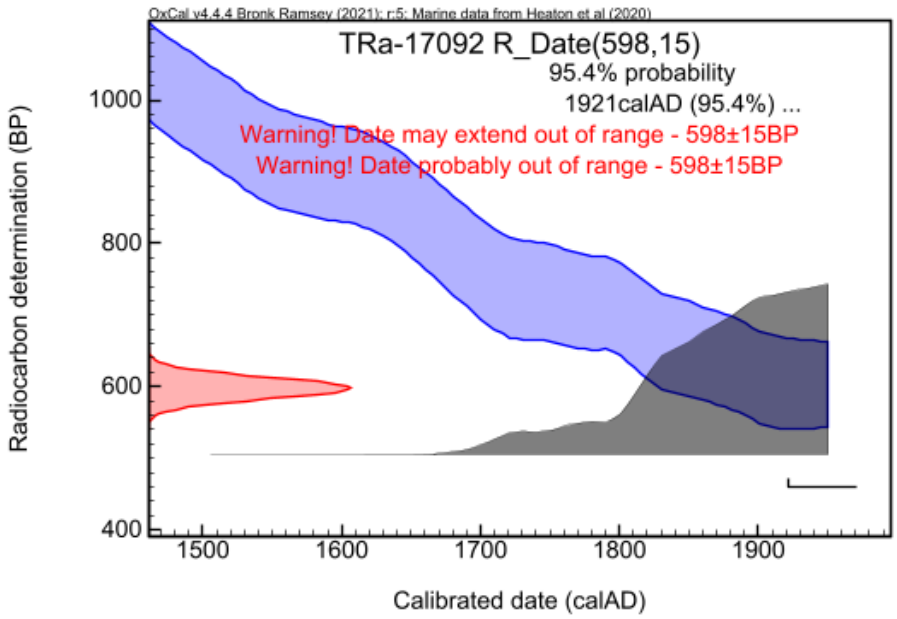

SID6922 lipid
SID7010 lipid
SID7013 lipid
SID7021 lipid
SID7134 lipid
Absorbance
Wavenumbers [cm-1]

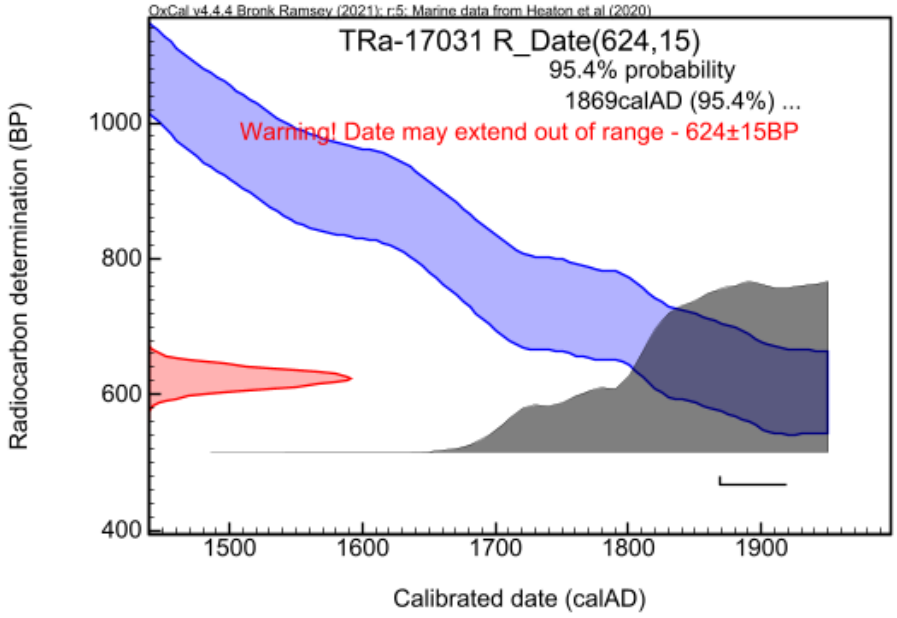

Parafilm
Pipette Bulb_rubber_New
Pipette Bulb_rubber_Old
2917 0.4103
2848 0.3135
1460 0.1368
1368 0.09711
1230 0.04469
1016 0.02177
719.4 0.1004
Absorbance
Wavenumbers [cm-1]

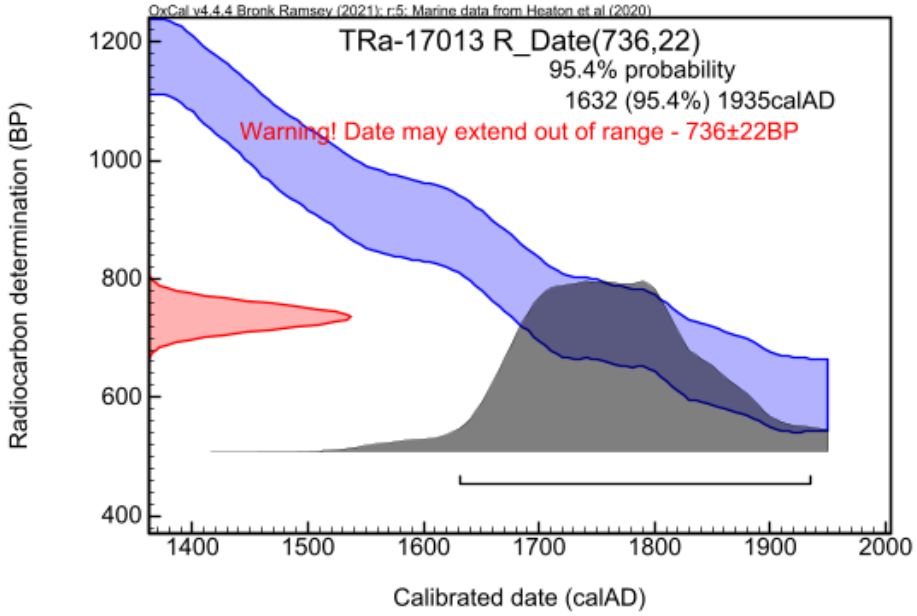

Parafilm
Pipette Bulb_rubber_New
Pipette Bulb_rubber_Old
Absorbance
Wavenumbers [cm⁻¹]

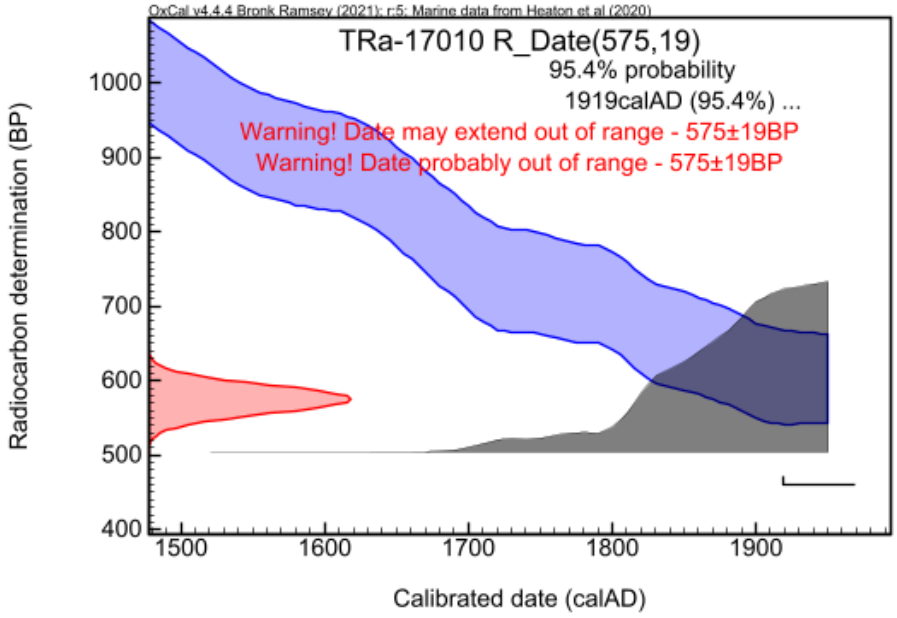
Appendix 8 Collagen results calibrated with Marine20

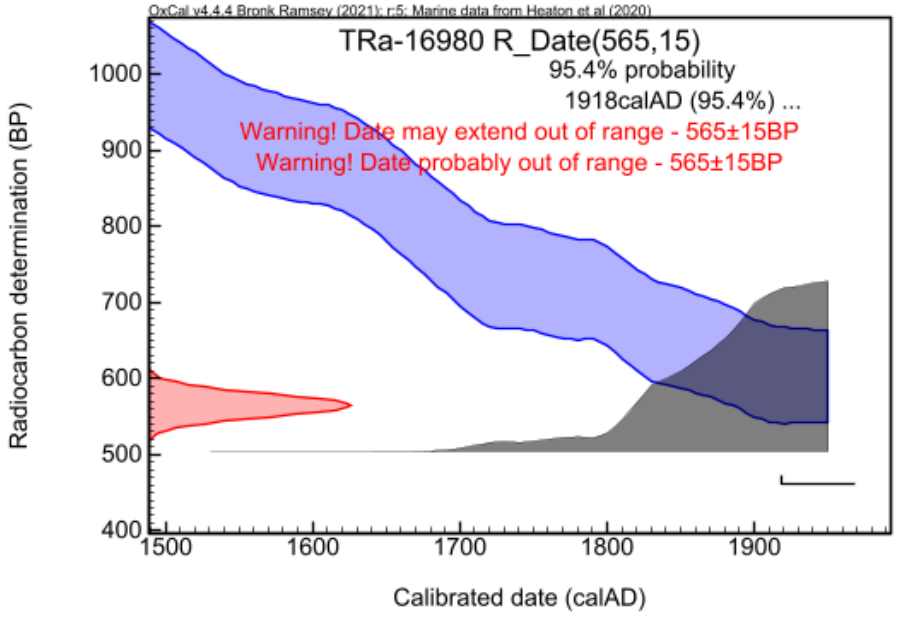


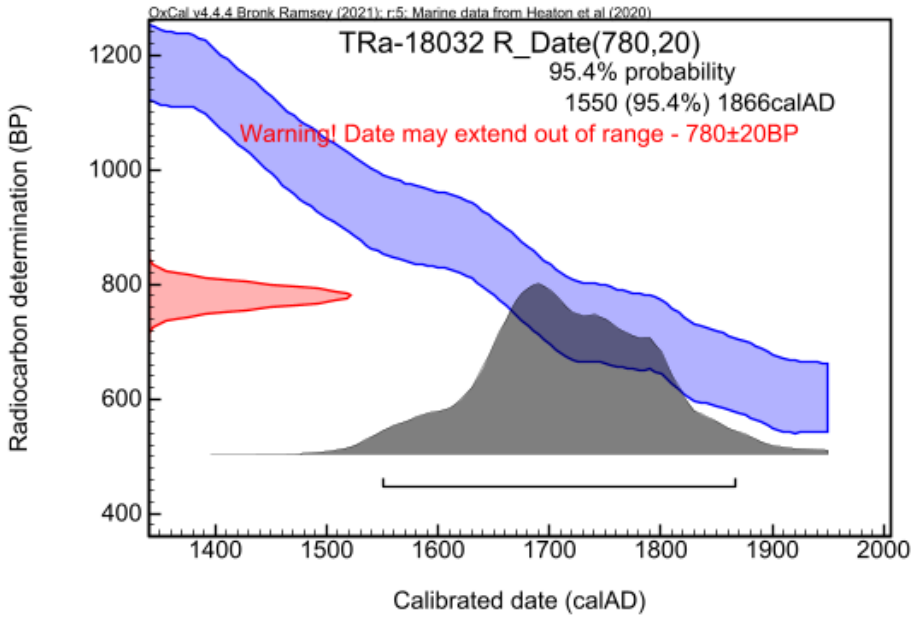


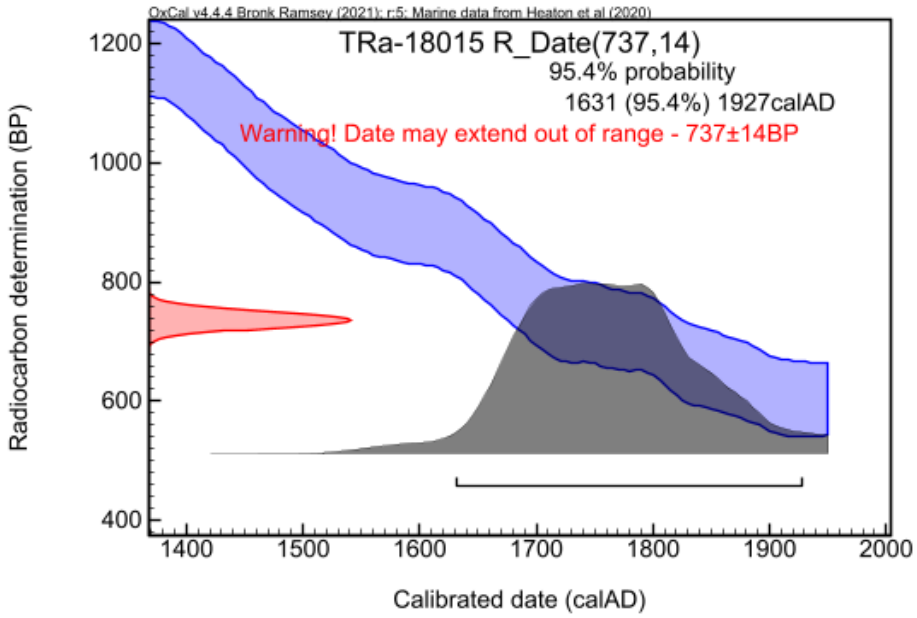

OxCal v4.4.4 Bronk Ramsey (2021); r:5; Marine data from Heaton et al (2020)
TRa-17031 R_Date(624,15)
95.4% probability
1869calAD (95.4%) ...
Warning! Date may extend out of range - 624±15BP
Radiocarbon determination (BP)
1000
800
600
400
1500
1600
1700
1800
1900
Calibrated date (calAD)

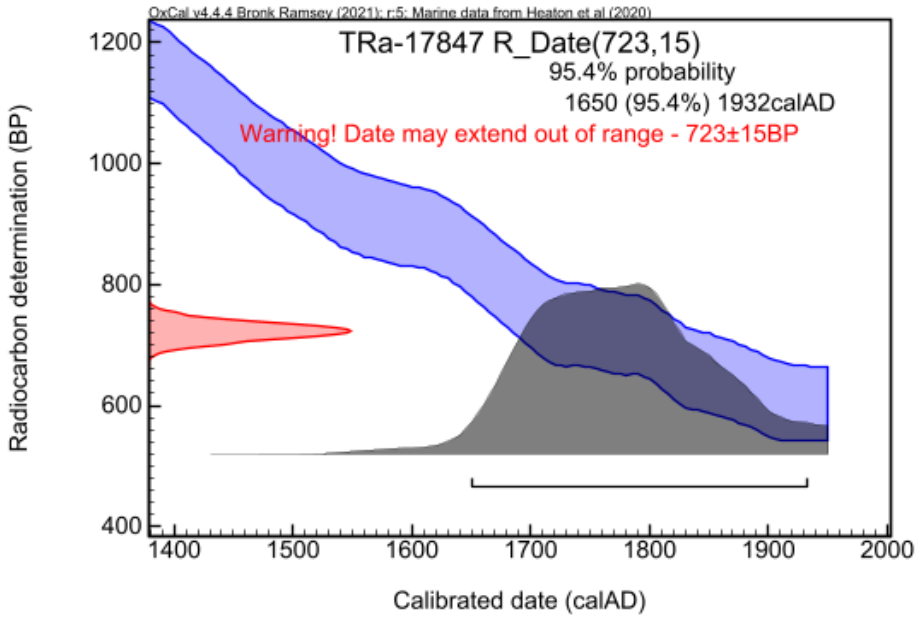

OxCal v4.4.4 Bronk Ramsey (2021); r:5; Marine data from Heaton et al (2020)
TRa-17013 R_Date(736,22)
95.4% probability
1632 (95.4%) 1935calAD
Warning! Date may extend out of range - 736±22BP
Radiocarbon determination (BP)
1200
1000
800
600
400
1400
1500
1600
1700
1800
1900
2000
Calibrated date (calAD)

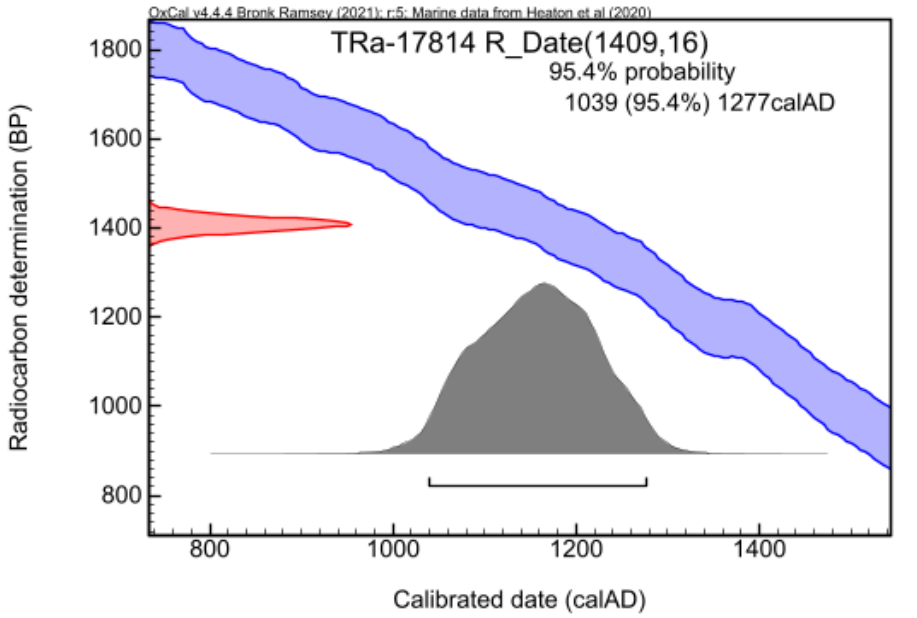

OxCal v4.4.4 Bronk Ramsey (2021); r:5; Marine data from Heaton et al (2020)
TRa-17010 R_Date(575,19)
95.4% probability
1919calAD (95.4%) ...
Warning! Date may extend out of range - 575±19BP
Warning! Date probably out of range - 575±19BP
Radiocarbon determination (BP)
1000
900
800
700
600
500
400
1500
1600
1700
1800
1900
Calibrated date (calAD)

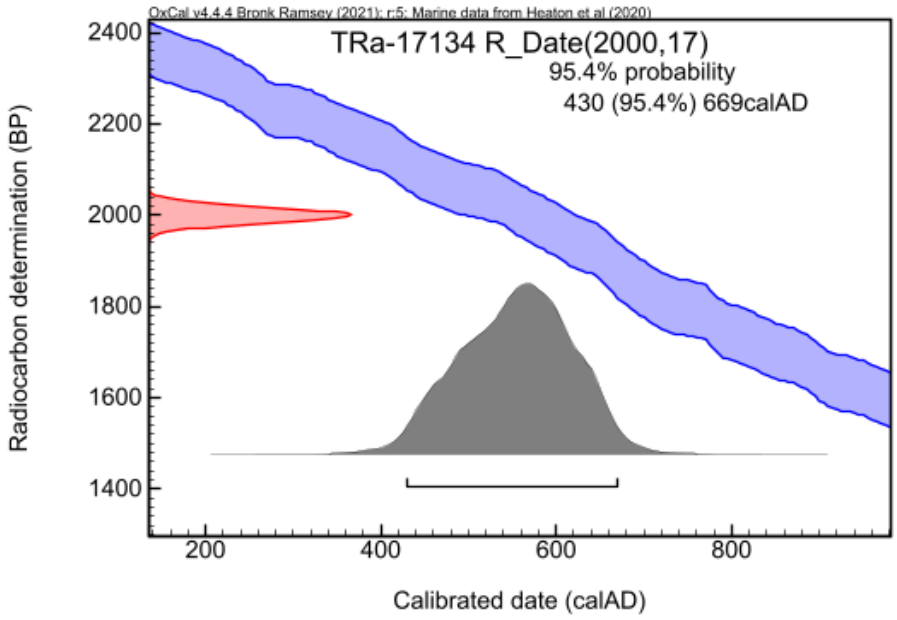

OxCal v4.4.4 Bronk Ramsey (2021); r:5; Marine data from Heaton et al (2020)
TRa-16980 R_Date(565,15)
95.4% probability
1918calAD (95.4%) ...
Warning! Date may extend out of range - 565±15BP
Warning! Date probably out of range - 565±15BP
Radiocarbon determination (BP)
1000
900
800
700
600
500
400
1500
1600
1700
1800
1900
Calibrated date (calAD)

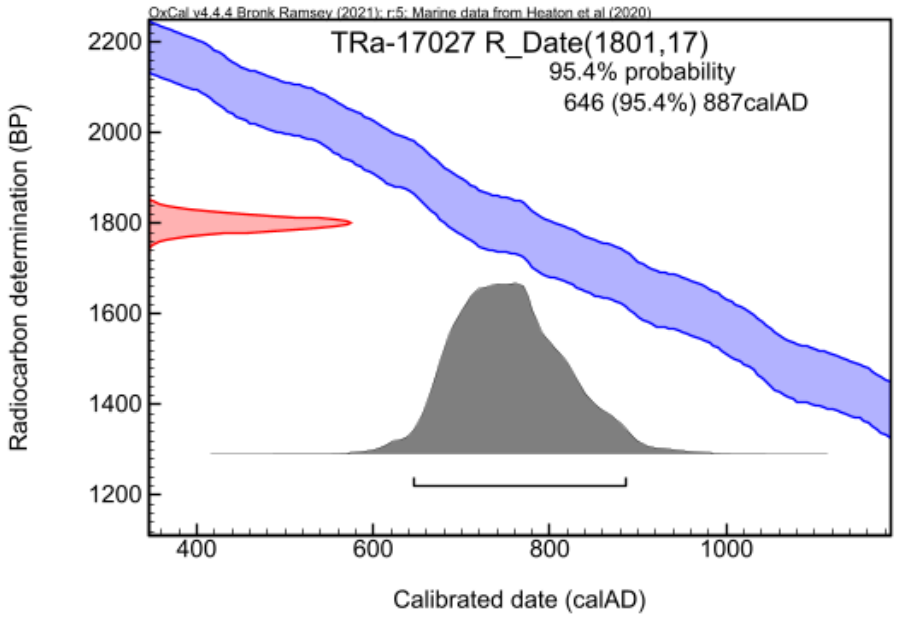

OxCal v4.4.4 Bronk Ramsey (2021); r:5; Marine data from Heaton et al (2020)
TRa-18032 R_Date(780,20)
95.4% probability
1550 (95.4%) 1866calAD
Warning! Date may extend out of range - 780±20BP
Radiocarbon determination (BP)
Calibrated date (calAD)
1200
1000
800
600
400
1400
1500
1600
1700
1800
1900
2000

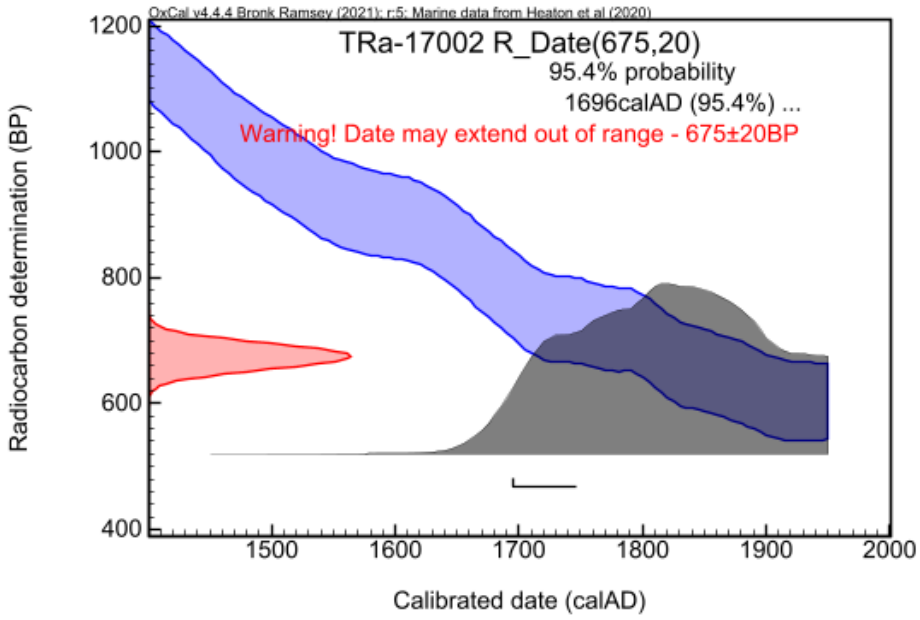

OxCal v4.4.4 Bronk Ramsey (2021); r:5; Marine data from Heaton et al (2020)
TRa-18015 R_Date(737,14)
95.4% probability
1631 (95.4%) 1927calAD
Warning! Date may extend out of range - 737±14BP
Radiocarbon determination (BP)
Calibrated date (calAD)
1200
1000
800
600
400
1400
1500
1600
1700
1800
1900
2000

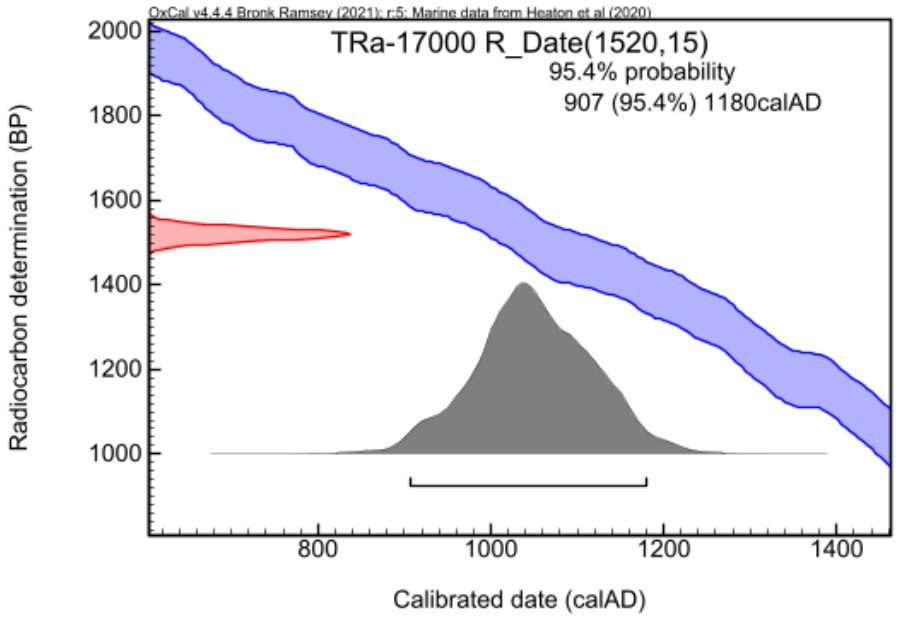

OxCal v4.4.4 Bronk Ramsey (2021); r:5; Marine data from Heaton et al (2020)
TRa-17847 R_Date(723,15)
95.4% probability
1650 (95.4%) 1932calAD
Warning! Date may extend out of range - 723±15BP
Radiocarbon determination (BP)
Calibrated date (calAD)

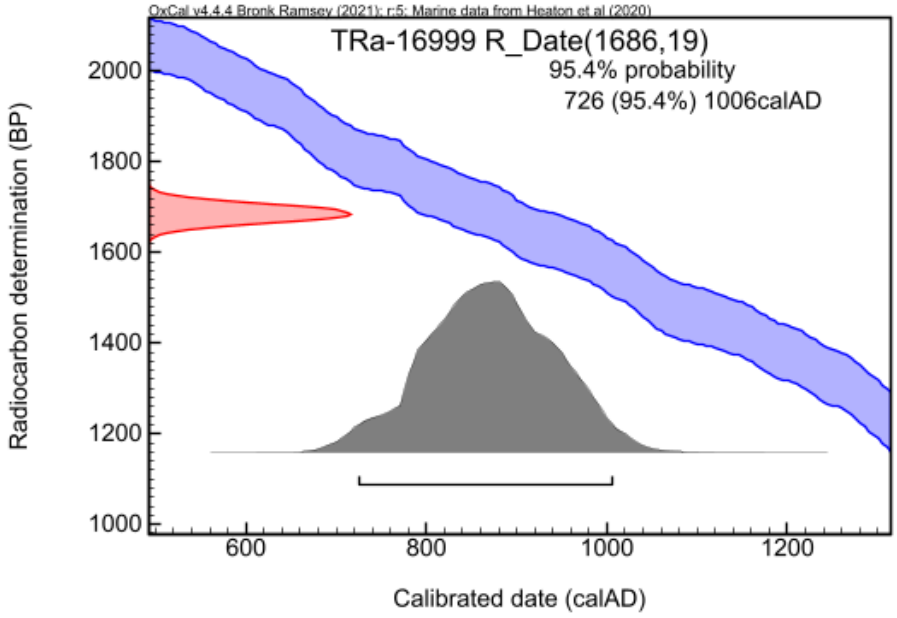

OxCal v4.4.4 Bronk Ramsey (2021); r:5; Marine data from Heaton et al (2020)
TRa-17814 R_Date(1409,16)
95.4% probability
1039 (95.4%) 1277calAD
Radiocarbon determination (BP)
Calibrated date (calAD)

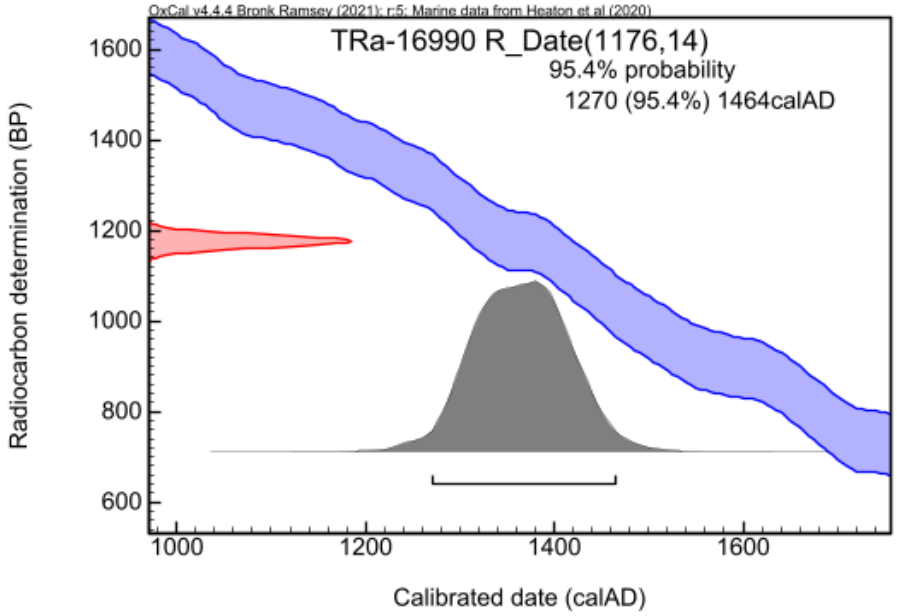

OxCal v4.4.4 Bronk Ramsey (2021); r:5; Marine data from Heaton et al (2020)
TRa-17134 R_Date(2000,17)
95.4% probability
430 (95.4%) 669calAD
Radiocarbon determination (BP)
2400
2200
2000
1800
1600
1400
200
400
600
800
Calibrated date (calAD)

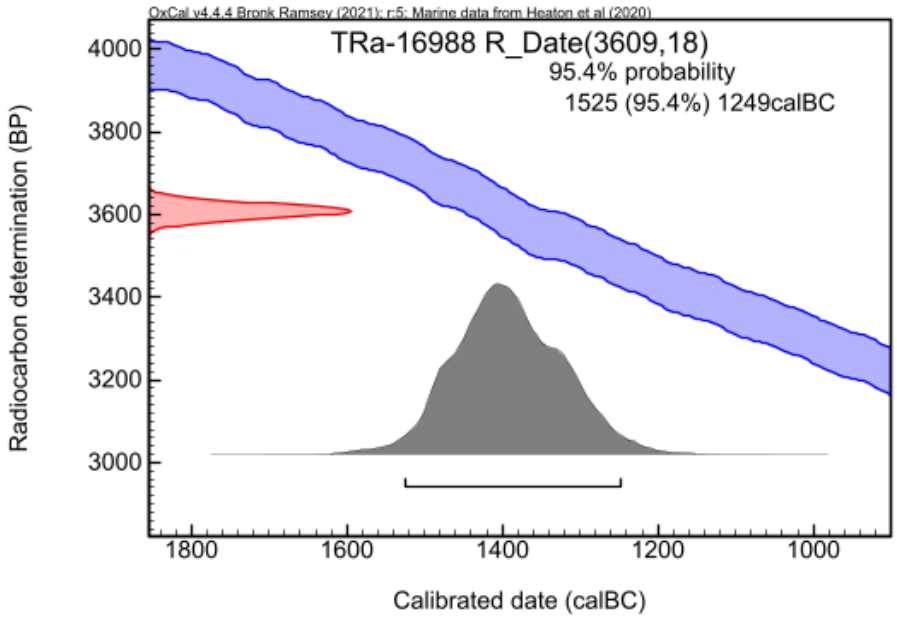

OxCal v4.4.4 Bronk Ramsey (2021); r:5; Marine data from Heaton et al (2020)
TRa-17027 R_Date(1801,17)
95.4% probability
646 (95.4%) 887calAD
Radiocarbon determination (BP)
2200
2000
1800
1600
1400
1200
400
600
800
1000
Calibrated date (calAD)

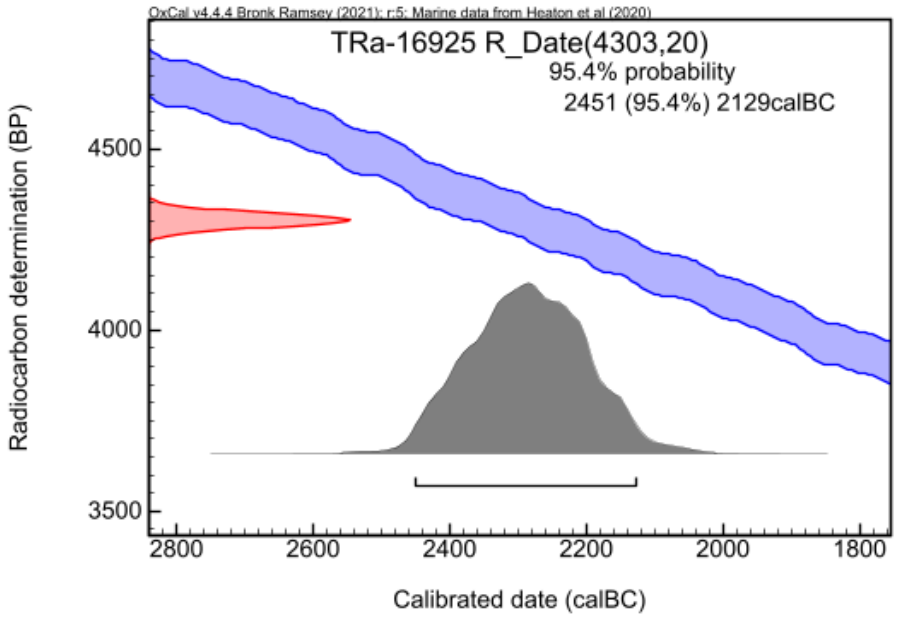

OxCal v4.4.4 Bronk Ramsey (2021); r:5; Marine data from Heaton et al (2020)
TRa-17002 R_Date(675,20)
95.4% probability
1696calAD (95.4%) ...
Warning! Date may extend out of range - 675±20BP
Radiocarbon determination (BP)
Calibrated date (calAD)

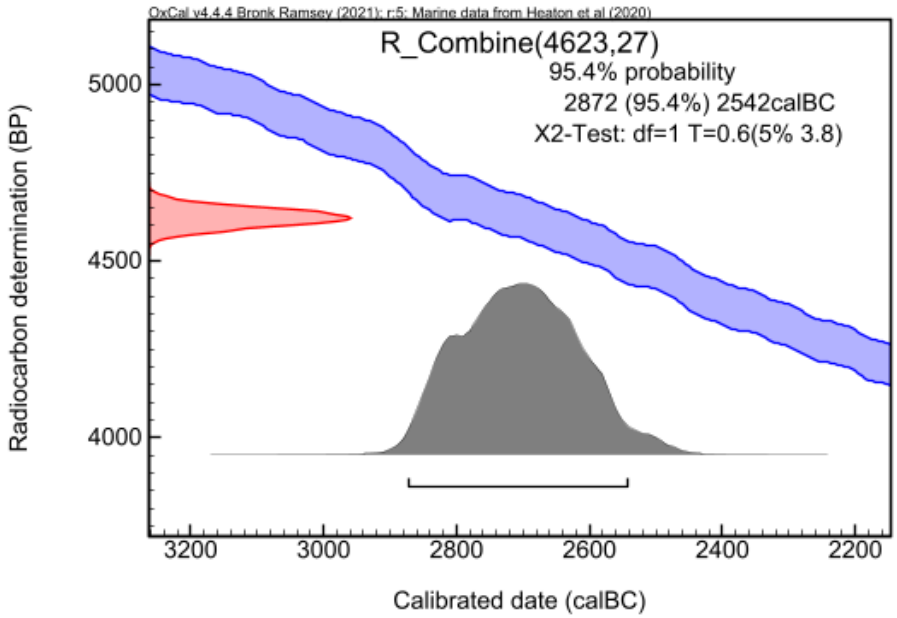

OxCal v4.4.4 Bronk Ramsey (2021); r:5; Marine data from Heaton et al (2020)
TRa-17000 R_Date(1520,15)
95.4% probability
907 (95.4%) 1180calAD
Radiocarbon determination (BP)
Calibrated date (calAD)


OxCal v4.4.4 Bronk Ramsey (2021); r:5; Marine data from Heaton et al (2020)
TRa-16999 R_Date(1686,19)
95.4% probability
726 (95.4%) 1006calAD
Radiocarbon determination (BP)
2000
1800
1600
1400
1200
1000
600
800
1000
1200
Calibrated date (calAD)

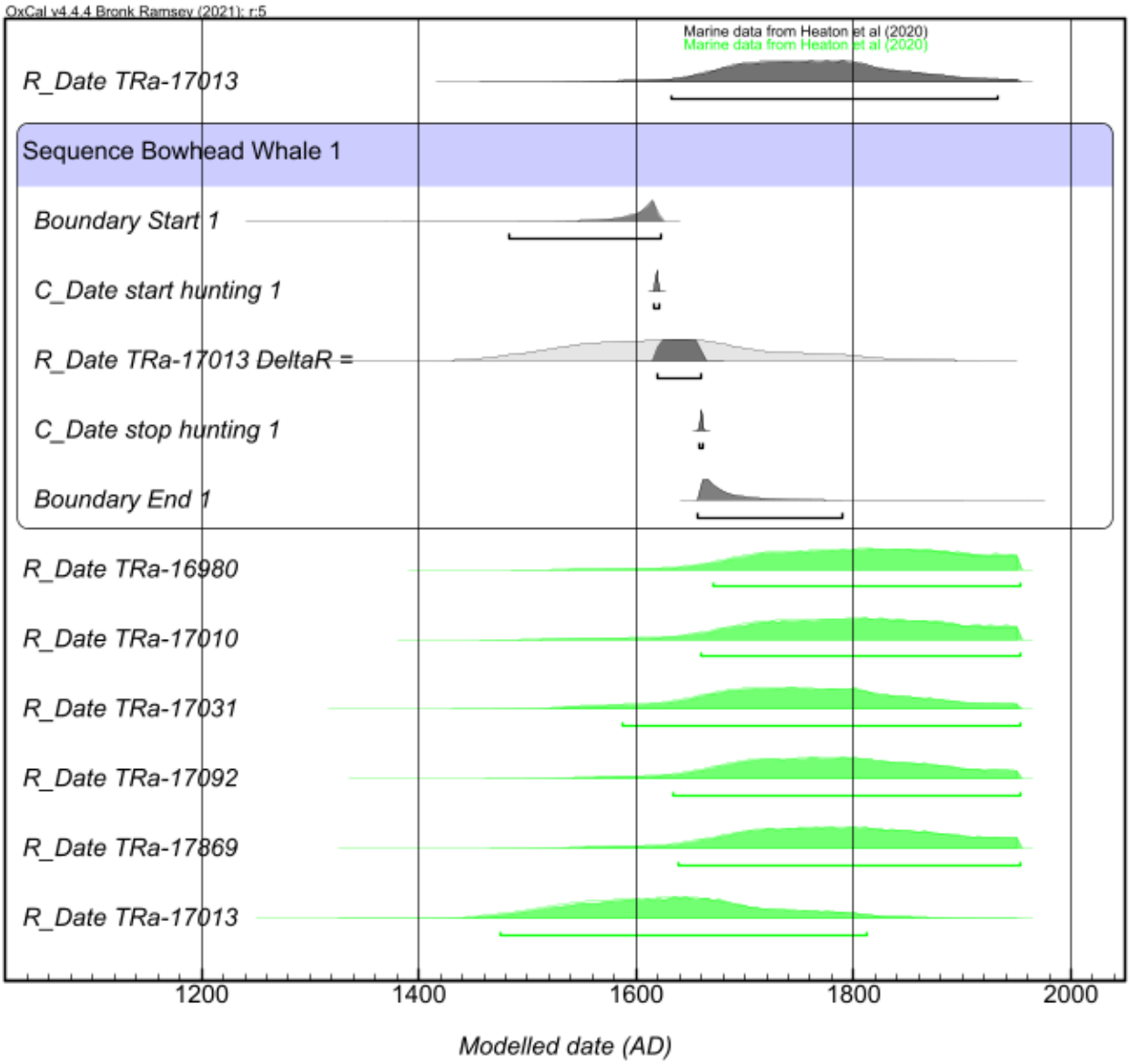

OxCal v4.4.4 Bronk Ramsey (2021); r:5; Marine data from Heaton et al (2020)
TRa-16990 R_Date(1176,14)
95.4% probability
1270 (95.4%) 1464calAD
Radiocarbon determination (BP)
1600
1400
1200
1000
800
600
1000
1200
1400
1600
Calibrated date (calAD)

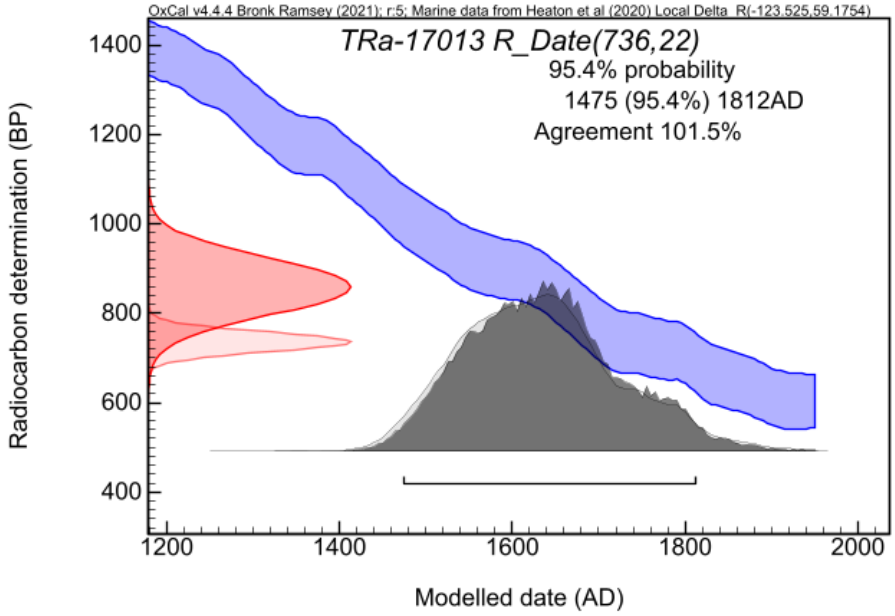

OxCal v4.4.4 Bronk Ramsey (2021); r:5; Marine data from Heaton et al (2020)
TRa-16988 R_Date(3609,18)
95.4% probability
1525 (95.4%) 1249calBC
Radiocarbon determination (BP)
4000
3800
3600
3400
3200
3000
1800
1600
1400
1200
1000
Calibrated date (calBC)

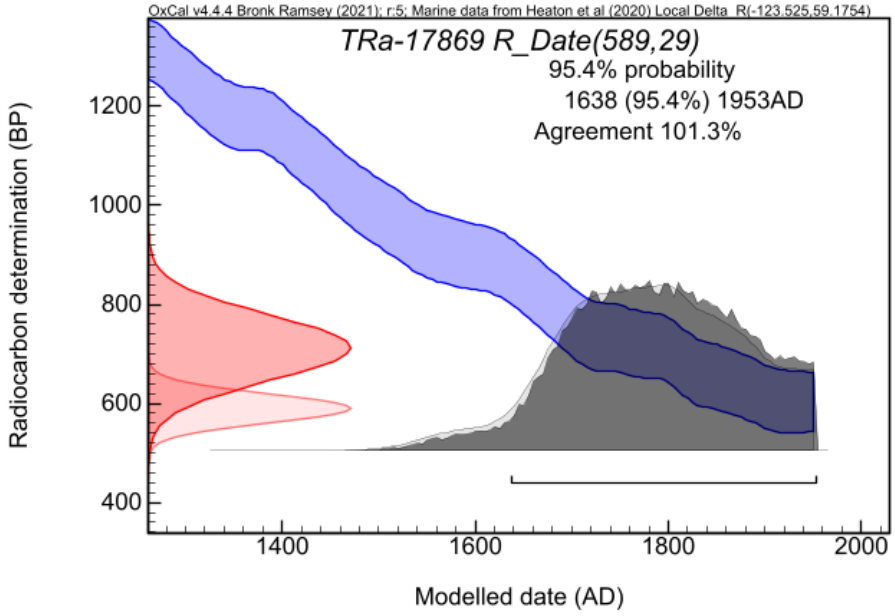

OxCal v4.4.4 Bronk Ramsey (2021); r:5; Marine data from Heaton et al (2020)
TRa-16925 R_Date(4303,20)
95.4% probability
2451 (95.4%) 2129calBC
Radiocarbon determination (BP)
4500
4000
3500
2800
2600
2400
2200
2000
1800
Calibrated date (calBC)

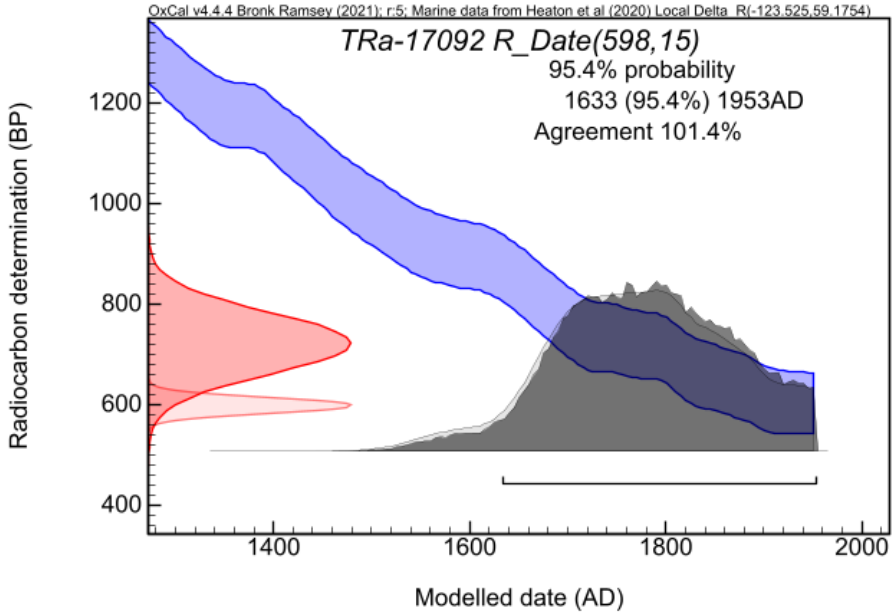

OxCal v4.4.4 Bronk Ramsey (2021); r:5; Marine data from Heaton et al (2020)
R_Combine(4623,27)
95.4% probability
2872 (95.4%) 2542calBC
X2-Test: df=1 T=0.6(5% 3.8)
Radiocarbon determination (BP)
5000
4500
4000
3200
3000
2800
2600
2400
2200
Calibrated date (calBC)

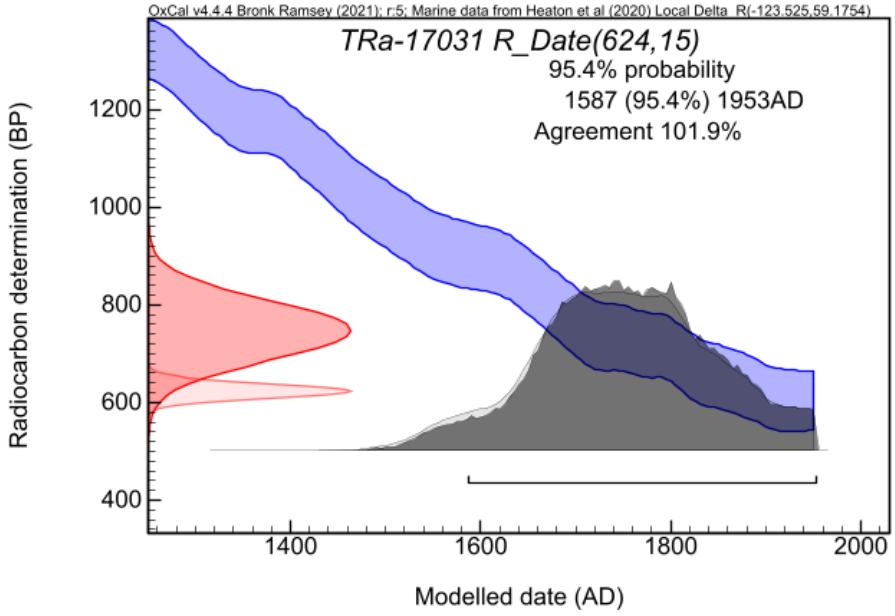
Appendix 9 DeltaR corrected dates

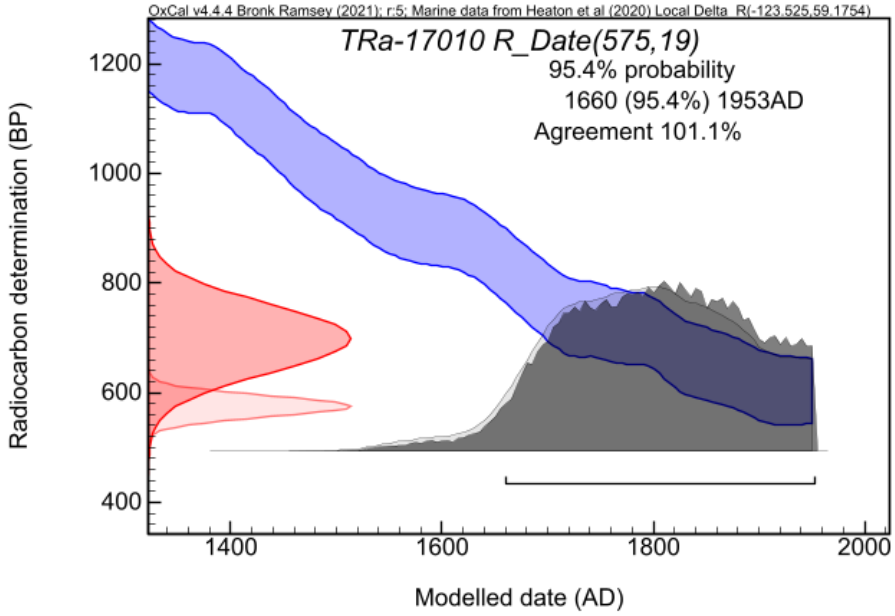


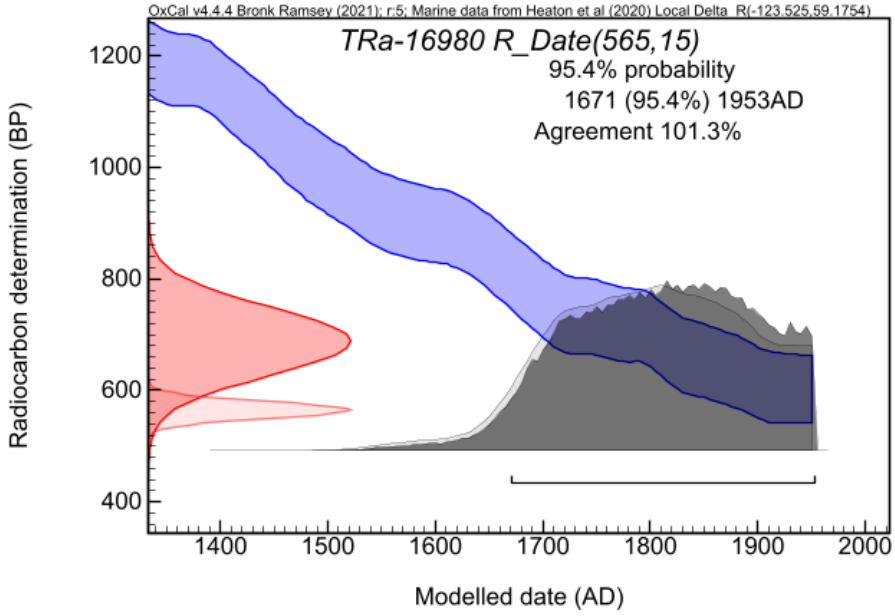

OxCal v4.4.4 Bronk Ramsey (2021); r:5; Marine data from Heaton et al (2020) Local Delta_R(-123.525,59.1754)
TRa-17013 R_Date(736,22)
95.4% probability
1475 (95.4%) 1812AD
Agreement 101.5%
Radiocarbon determination (BP)
Modelled date (AD)
1400
1200
1000
800
600
400
1200
1400
1600
1800
2000

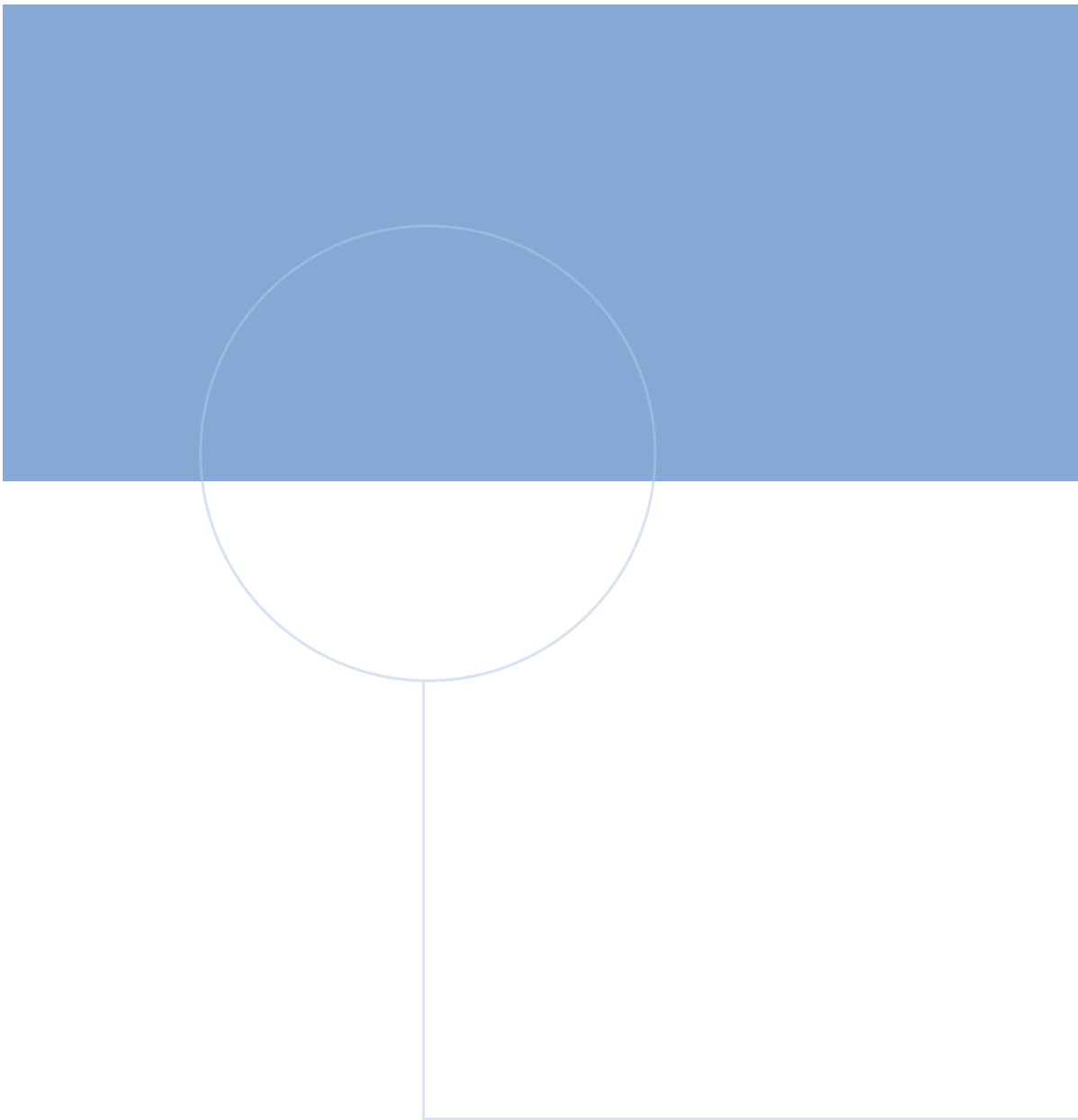

OxCal v4.4.4 Bronk Ramsey (2021); r:5; Marine data from Heaton et al (2020) Local Delta_R(-123.525,59.1754)
TRa-17869 R_Date(589,29)
95.4% probability
1638 (95.4%) 1953AD
Agreement 101.3%
Radiocarbon determination (BP)
Modelled date (AD)
1200
1000
800
600
400
1400
1600
1800
2000


OxCal v4.4.4 Bronk Ramsey (2021); r:5; Marine data from Heaton et al (2020) Local Delta_R(-123.525,59.1754)
TRa-17092 R_Date(598,15)
95.4% probability
1633 (95.4%) 1953AD
Agreement 101.4%
Radiocarbon determination (BP)
1200
1000
800
600
400
1400
1600
1800
2000
Modelled date (AD)


OxCal v4.4.4 Bronk Ramsey (2021); r:5; Marine data from Heaton et al (2020) Local Delta_R(-123.525,59.1754)
TRa-17031 R_Date(624,15)
95.4% probability
1587 (95.4%) 1953AD
Agreement 101.9%
Radiocarbon determination (BP)
1200
1000
800
600
400
1400
1600
1800
2000
Modelled date (AD)


OxCal v4.4.4 Bronk Ramsey (2021); r:5; Marine data from Heaton et al (2020) Local Delta_R(-123.525,59.1754)
TRa-17010 R_Date(575,19)
95.4% probability
1660 (95.4%) 1953AD
Agreement 101.1%
Radiocarbon determination (BP)
1200
1000
800
600
400
1400
1600
1800
2000
Modelled date (AD)


OxCal v4.4.4 Bronk Ramsey (2021); r:5; Marine data from Heaton et al (2020) Local Delta_R(-123.525,59.1754)
TRa-16980 R_Date(565,15)
95.4% probability
1671 (95.4%) 1953AD
Agreement 101.3%
Radiocarbon determination (BP)
1200
1000
800
600
400
1400
1500
1600
1700
1800
1900
2000
Modelled date (AD)



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