RADIOCARBON DATING MARINE SHELL: CHALLENGES AND OPPORTUNITIES IN CANADIAN COASTAL ARCHAEOLOGY

by © Marisa Aimee Dusseault

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ABSTRACT

Marine mollusk shells are the most abundant materials found at coastal archaeology sites. They are often found in large accumulations called middens which are formed as a result of shellfish processing activities and may be features of dwelling sites. Despite the abundance of marine mollusk shells at coastal sites, terrestrial samples are often favoured for radiocarbon analysis because they are free from local marine reservoir effects. In this thesis, I investigate intra-shell radiocarbon variability in three mollusk species from coastal sites across Canada: *Saxidomus gigantea* from British Columbia, *Mya arenaria* from Nova Scotia, and *Crassostrea virginica* from Prince Edward Island. Each of these species has different growth strategies that must be considered to understand time-averaging effects in the radiocarbon measurements. Short-lived *C. virginica* samples from Prince Edward Island had the best agreement between intra-shell radiocarbon measurements. Additional radiocarbon measurements on twelve *M. arenaria* shells from Port Joli Harbour, Nova Scotia show good agreement with the previously published terrestrial dates and paired marine-terrestrial samples are used to calculate the first archaeological ΔR values for the region. Building confidence in marine shell radiocarbon will greatly widen the number of samples that archaeologists can study from coastal midden sites, especially ones that may be rapidly eroding and lack terrestrial samples.

GENERAL SUMMARY

People living in coastal regions have harvested clams, mussels, and oysters for food for thousands of years. The hard shells of these creatures that were harvested hundreds if not thousands of years ago can still be found today in many coastal regions. These shells contain carbon, which archaeologists analyse to determine when the mollusk shell was formed and in turn when people harvested the mollusks. What makes analyzing carbon from shells challenging is that they grow like trees - forming lines with each year of growth - incorporating carbon from the ocean as they grow. The concentration of carbon in the ocean can also change across time and space meaning that the carbon signal can change throughout the mollusk's life, resulting in portions of the shell appearing older than they actually are. In this thesis I study shells from different species and different coastal locations within Canada to show that some species are easier for archaeologists to analyze than others. This depends on the age of the mollusk and the local marine environment. I also use radiocarbon dates from shells from Port Joli Harbour Nova Scotia, a harbour which has been occupied for thousands of years, to show that dates from marine shell agree well with dates from wood and bone and suggest a value that can be used to correct for changes in the local marine environment, making the dates more accurate.

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I acknowledge that the samples studied in this thesis are from the lands of the Coast Salish and Shíshálh people of British Columbia and the Mi’kmaw people of Nova Scotia and Prince Edward Island and express my thanks for the opportunity to study archaeological materials from their lands. I also acknowledge that the lands on which this research was conducted are situated in the traditional territories of diverse Indigenous groups, and I acknowledge with respect the diverse histories and cultures of the Beothuk, Mi’kmaq, Innu, and Inuit of the province of Newfoundland.

I would also like to thank the School of Graduate Studies at Memorial University and NSERC for providing the funds to complete this research, and especially to Dr. Meghan Burchell, the Archaeology Department, and the rest of the Burchell Lab Group who have been very flexible and gracious with me even as I worked on this project part-time. My time with the Burchell Lab Group will always be deeply cherished.

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ABBREVIATIONS

yrs BP	years before present (before 1950 AD)
calBP	calibrated years before present (1950 AD)
calBC/AD	calibrated years in BC or AD
^{14}C	the radioactive carbon isotope, used interchangeably with radiocarbon throughout
F14C	fraction of modern carbon
FT-IR	Fourier Transform Infrared Spectroscopy
PJH	Port Joli Harbour
ΔR	local marine reservoir offset

CHAPTER 1

INTRODUCTION

1.1 INTRODUCTION TO RADIOCARBON DATING, SHELL MIDDENS, AND A FEMINIST APPROACH TO RESEARCH

Shell middens are unique lenses into coastal archaeological sites that allow archaeologists to interpret past climates, resource management strategies, and human-environment interactions (M. W. Betts, 2019; Cannon & Burchell, 2009; Carlson et al., 2017). Shell midden archives, while extremely useful study sources for archaeologists, often pose challenges in the form of difficult stratigraphy and site-formation reconstruction while simultaneously offering up an abundance of materials to be radiocarbon dated in the form of biogenic marine calcium carbonates ($CaCO_3$) from mollusk shells (Claassen, 1998). However, radiocarbon dating these materials is not straightforward as mollusk shell radiocarbon is affected by the local marine reservoir offsets, the hard water effect, and the old shell problem (Douka et al., 2010). These archaeological carbonate archives offer up many challenges and opportunities which have not been fully realized in Canadian coastal archaeology.

This thesis is composed of two main chapters. The first explores intra-shell radiocarbon variability from three species of mollusks from archaeological sites British Columbia, Prince Edward Island, and Nova Scotia to investigate how the growth strategies of different mollusks species should inform sampling strategies and what consequences intra-shell radiocarbon variability may have on chronology construction and climate reconstruction. The second, focuses entirely on Port Joli Harbour, Nova

Scotia to investigate effects of incorporating marine dates from the soft-shell clam, *Mya arenaria*, into the existing terrestrial radiocarbon chronology and report the first archaeological local marine reservoir values for the harbour.

In this chapter, I outline my theoretical approach to radiocarbon dating, which incorporates a feminist perspective, and a critical approach to the archaeological use of time and chronologies. I also discuss the measurement and calibration strategies for radiocarbon dating marine samples as well as the details of radiocarbon dating marine shells, which includes discussions on shell growth and its relationship to the local marine ^{14}C environment. Lastly, I elaborate on the status of radiocarbon dating, and specifically marine radiocarbon dating, in Canadian Archaeology.

1.2 RE-THINKING RADIOCARBON: A FEMINIST APPROACH TO UNDERSTANDING TIME

Discussions surrounding the issue of the imposition of linear time on the past have prompted wider discussions about privilege in archaeology: who gets to study the past, and who gets to write the history of the past? By adopting a feminist approach, we can question power structures in the discipline, but especially those regarding which samples are selected, and how radiocarbon data is analyzed and used (Kincaid et al., 2007; Wylie, 2007). Shellfish harvesting was and is an activity completed primarily by women and the discipline of archaeology has historically placed less significance on the stories of women (Dahlberg, 1981). Excluding these materials from archaeological studies and from chronologies undermines their value in the archaeological record and excludes them from a significant portion of archaeological literature.

It is undeniable that radiocarbon dating is one of the most powerful tools available to archaeologists. The ability to take an ordinary object or material from an archaeological deposit and be able to determine a chronometric date for when it was deposited into that context has transformed the way archaeologists approach time and its importance to the discipline, but the way in which archaeologists have used and will continue to use radiocarbon is not static. Bayliss (2009) describes three radiocarbon revolutions that have taken place within archaeology: the first revolution is linked to the initial establishment of the scientific principles of radiocarbon dating in 1949, the second revolution was ignited by the need for dates to be calibrated, and the third revolution is characterized by the use of Bayesian model to build chronologies and the wide availability of software to do such analysis. These technical advances have undoubtedly contributed immensely to the field and expanded the types of questions that can be asked and how they are answered, but the intersection of these technical advances with growing discussions around ‘slow archaeology’ and values from the feminist approach may lead to further shifts in the use of radiocarbon data, who collects it, and how it is presented (Caraher, 2019).

Historically and presently, the dominant approach to understanding time in archaeology is the culture-historical framework. This theoretical framework focuses on segmenting time into periods with characteristic attributes, such as ceramics or dwelling styles, and uses those constructed periods to draw comparisons with other cultures and construct a timeline of cultural evolution (Trigger, 2006). Radiocarbon data is often used to uphold the culture-historical framework as it tends to be used to construct chronologies and create boundaries between these cultural periods.

When used at their best, chronologies can reinforce a sense of belonging of local Indigenous populations to their lands, but when used at their worst, they are used to emphasize the culture-historical framework by focusing heavily on arbitrary temporal periods, novelty, and origins, and takes the control of the past away from those to whom it rightfully belongs (P. Steeves, 2020). The lens of the culture-historical approach tends to keep western culture as the focus, as it places the arrival of modern western civilization as the culmination of a series of increasingly more "complex" social structures (Carlson et al., 2017; P. F. Steeves, 2015). Despite these critiques of the culture-historical approach, organizing time periods into neat little boxes and representing thousands of years of occupation as a series of single data points is simple and intuitive and often makes comparisons between arbitrary time periods easy and seemingly meaningful (Dempsey, 2008).

As we critically reconsider the ways archaeologists acquire, analyze, incorporate, and interpret dates, new histories are emerging. For example the groundbreaking work by Paulette Steeves has shifted the focus to ask what role Indigenous people have in the culture-historical approach, the Culture History approach often overlooks the needs and wants of Indigenous groups and offers them information that they already know: that they have lived here forever (P. Steeves, 2020; P. F. Steeves, 2015). The lack of data transparency in North American archaeology only widens the disconnect between those who collect radiocarbon dates and those whose pasts are being interpreted with these data. The feminist approach taken here considers this disconnect between those who collect radiocarbon dates and those to whom it carries significance and places a high priority on data transparency and critically questioning radiocarbon data.

Although many feminist archaeologists explicitly focus on research questions regarding gendered experiences, at its core the feminist approach examines the role of power structures in archaeology, and radiocarbon studies operate within a power structure in archaeology (Wylie, 2007). This is exaggerated in North America where most radiocarbon labs are a commercial industry whereby the researcher sends samples to the lab with varying degrees of pre-processing or screening, and a calibrated date is returned for a price. This for-profit model inhibits researchers from being involved at every step of the analysis process, creating a black-box problem. This structure shapes the types of research questions that are proposed. The feminist approach taken here emphasized a need for transparency at all levels of the radiocarbon dating process to make it more inclusive and accessible, but also to encourage researchers to incorporate archeological shellfish archives – an archaeological record produced primarily by women – into their research questions.

Radiocarbon measurements are messy, disordered, constantly being revised, and have greater uncertainties than those reported by the final calibrated age ranges. Adopting a feminist approach encourages more rigorous questioning of radiocarbon data and understanding where this variability comes from, how it is related to the samples themselves, the sampling strategies used, and geographic and oceanographic factors. Considering these uncertainties also requires an interdisciplinary approach whereby there is deep collaboration between physicists and archaeologists. This has been very successful in many European institutions (see Lev et al., 2022, Regev et al., 2020, Wacker et al., 2010). Transparent labs and inquisitive archaeologists who are willing to get their hands dirty in the math will be rewarded with more than exhausting, ladder-like chronologies, but rather a deeper understanding of the interconnectedness of archaeological materials, environment, and time. A comprehensive view of radiocarbon offers up more than single dates in time to be plotted and pinpoint events in time, but instead a more complete and sometimes challenging view of how seasonal, decadal, and centennial cycles in the ocean and atmosphere affect our understanding of time. For archaeologists, this use of radiocarbon is unconventional and pushes back against the power structure in place that upholds the culture-historical approach which is highly focused on the oldest and the most novel findings.

1.3 RADIOCARBON AND THE CALIBRATION OF DATES FROM MARINE SHELL

The world's oceans act as a carbon reservoir. The levels of carbon isotopes ^{12}C,^{13}C and ^{14}C — with ^{12}C and ^{13}C being stable isotopes and ^{14}C being an unstable radioactive isotope — maintain a roughly constant distribution throughout the Earth's atmosphere over time. Living things are in equilibrium with their respective carbon resources throughout their lives and have predictable offsets from these resources. As ^{14}C is an unstable isotope, it will decay into ^{14}N by converting a neutron to a proton at a known rate called a half-life. This decay constantly occurs, but the concentration of 14C is replenished in living organisms because they are in exchange with their environment. However, when living things die, the radioactive carbon isotope ^{14}C will occur without replenishment. As the decay rate of ^{14}C is known, this basic principle allows for the determination of time since death by analyzing the ratio of $^{14}C/^{12}C$ or $^{14}C/^{13}C$ (Arnold & Libby, 1949; Libby, 1961).

This process has since been refined to account for fluctuations in ^{14}C in the environment over time by using calibration curves. These curves map the ^{14}C signatures in the environment over time using samples with known ages like tree rings and corals (P. J. Reimer et al., 2009, 2013, 2020). The radiocarbon calibration process results in a probability distribution for the final dates. An example of a radiocarbon date calibration is shown in Figure 1.1. Radiocarbon dates are often reported in years before 1950 AD, which is considered as "present" in radiocarbon convention (Stuiver & Polach, 1977). Analyzing samples originating from after 1950 require a different approach due to the spike in atmospheric and marine ^{14}C during testing of nuclear weapons in the 1950's and 1960's (Reimer et al., 2004; Scourse et al., 2012, Brock et al., 2018).

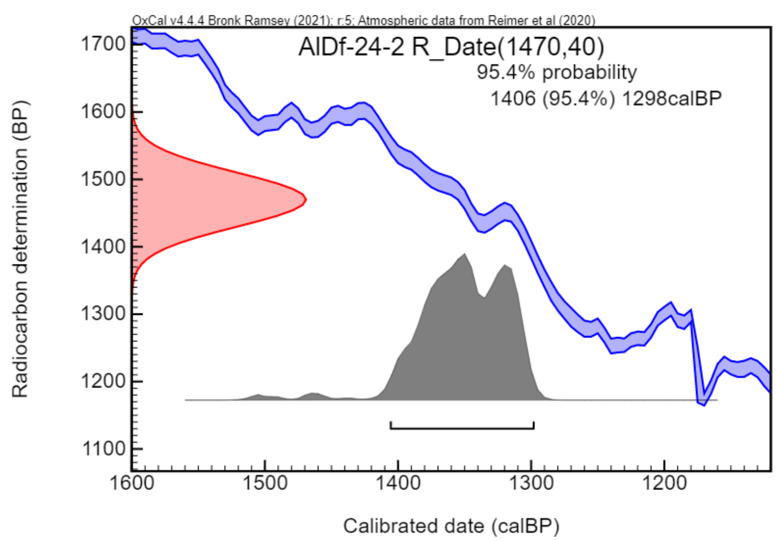

Figure 1.1 Example of a radiocarbon date calibration showing the uncalibrated age measurement on the y-axis, the atmospheric calibration curve in blue, and the calibrated probability distribution on the x-axis.

Many processes in nature result in the preferential uptake of specific isotopes over others due to their different masses. Any process in which this preferential uptake occurs is called a fractionation process. This process results in the isotopic carbon signatures of the organisms being different than the environment from which these isotopes were incorporated (Fahrni et al., 2017; Stuiver & Polach, 1977). Fractionation can be accounted for by correcting sample activities before they are converted to a conventional age by normalizing the $\delta^{13}C$ sample measurements to -25 parts per mil with respect to the Pee Dee Belemnite standard (Crann et al., 2017; Stuiver & Polach, 1977). From this normalized sample activity, a conventional radiocarbon age can be calculated using the known half-life of ^{14}C.

For marine mollusks, their carbon signatures are primarily derived from the marine environments in which they live, which contain little atmospheric carbon. Carbon is incorporated into the seawater in the form of dissolved CO_2 and HCO_3 (Douka et al., 2010). Due to the uneven mixing of carbon between the atmosphere and oceans, carbon may become locked deep in the ocean for lengths of time much longer than that of the half-life of radiocarbon resulting in more ^{14}C depletion and thus an older apparent age (P. J. Reimer et al., 2002; R. W. Reimer & Reimer, 2017). Depending on specific local marine conditions like upwelling and downwelling — systems that transport deep ocean water to and from surface waters — radiocarbon ages of mollusks may appear significantly older than terrestrial samples of the same age (Douka et al., 2010; R. W. Reimer & Reimer, 2017).

On average, the global marine reservoir has an apparent age 400 years older than that of the atmosphere and this value is refined every few years with the publication of updated marine and terrestrial calibration curves (Heaton, Blaauw, et al., 2020; Heaton, Köhler, et al., 2020). This average global offset between marine and terrestrial carbon reservoirs is accounted for by the use of an appropriate marine calibration curve when calibrating marine radiocarbon dates, however local ocean upwelling patterns, input of freshwater runoff or other inputs of old carbon such as limestone leaching results in spatial variations in this offset (Douka et al., 2010). A correction that accounts for more variability in the marine carbon reservoir is required for marine samples and is called the local marine reservoir offset (ΔR). A positive ΔR value indicates an older reservoir age when calibrating marine radiocarbon dates, a positive ΔR value will shift the calibration to a younger portion of the calibration curve. An example of this is shown in Figure 1.2.

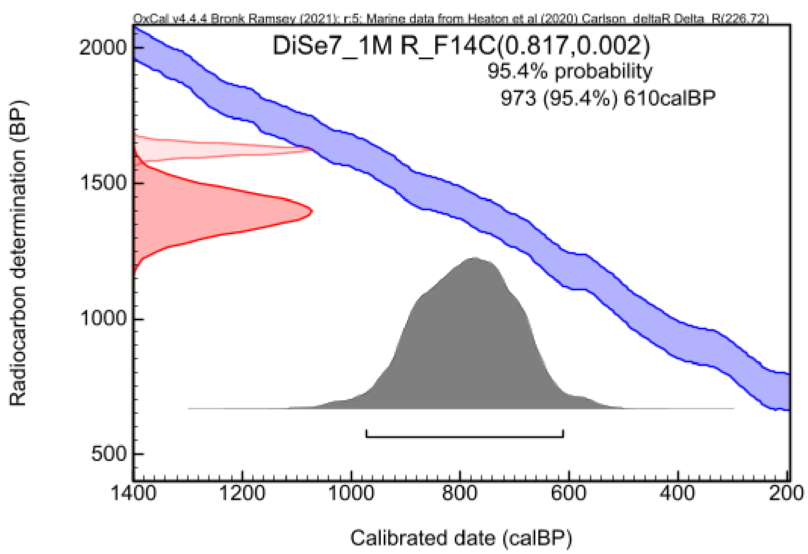

Figure 1.2 Example of a marine radiocarbon calibration showing the uncalibrated measurement (light pink) and its shifted value due to the ΔR correction (dark pink) on the y-axis, the marine calibration curve in blue, and the calibrated age range on the x-axis.

To calculate a ΔR value, an uncalibrated marine age is needed and either a contemporaneous calibrated terrestrial radiocarbon date or a known calendar year of collection. In archaeological applications, contemporaneous marine and terrestrial radiocarbon dates are often used. This dataset — a marine radiocarbon measurement and a calibrated terrestrial radiocarbon measurement from the same context — is called a marine-terrestrial pair. By reverse calibrating the calibrated terrestrial age to the marine calibration curve, an expected marine age can be determined (R. W. Reimer & Reimer, 2017). The difference between the expected marine age and the actual measured age is the ΔR value. This calculation is discussed in more detail in Chapter 3.

The measurement and application of the most suitable ΔR correction for any given coastal archaeology site can be challenging, and this becomes more challenging when patterns of shell growth, sampling, and ΔR variability are considered. These challenges are discussed in the next section.

1.4 ΔR, SHELL GROWTH, AND INTRA-SHELL VARIABILITY

In archaeological applications ΔR is necessary for proper calibration of marine radiocarbon dates and because ΔR is intricately linked with oceanographic patterns and climate change, studying spatial and temporal changes in ΔR can allow for interpretations of human-environment interactions in the context of broader trends in climate (Andrus et al., 2005; Hadden et al., 2023; Sandweiss et al., 2020; Sherwood et al., 2008). When calibrating marine radiocarbon dates, it is important to use a spatially local ΔR value for the site in question. If the ΔR was calculated using a marine-terrestrial pair from a site hundreds of kilometers away, even if it is a securely dated pair it may not be an acceptable value for the site in question. Sub-regional ΔR values are favourable for archaeological studies because they can provide a higher degree of chronological accuracy(Hadden et al., 2023; Lindauer et al., 2022; Rick et al., 2012).

Additional care is required to understand how the spatial and temporal trends in ΔR are combined with the growth strategies of marine mollusks. The carbon isotopes in the $CaCO_3$ biomineralized by marine mollusks reflect those of their dissolved inorganic carbon (DIC) source. The isotopic signatures of their marine DIC source may change over time as freshwater input varies or ocean current systems strengthen or weaken. Further, mollusks deposit subsequent layers of carbonates in their shells as they grow, and form both seasonal and annual growth bands, similar to how trees form growth rings (see Figures 1.3 and 1.4). Thus, the carbon isotopic makeup of these growth bands will reflect that of the DIC source at the time of biomineralization meaning that the ^{14}C signatures within these mollusk shells may also be subject to the same, seasonal, annual, and decadal variations in the local marine reservoir that affect the ^{14}C levels in the shell

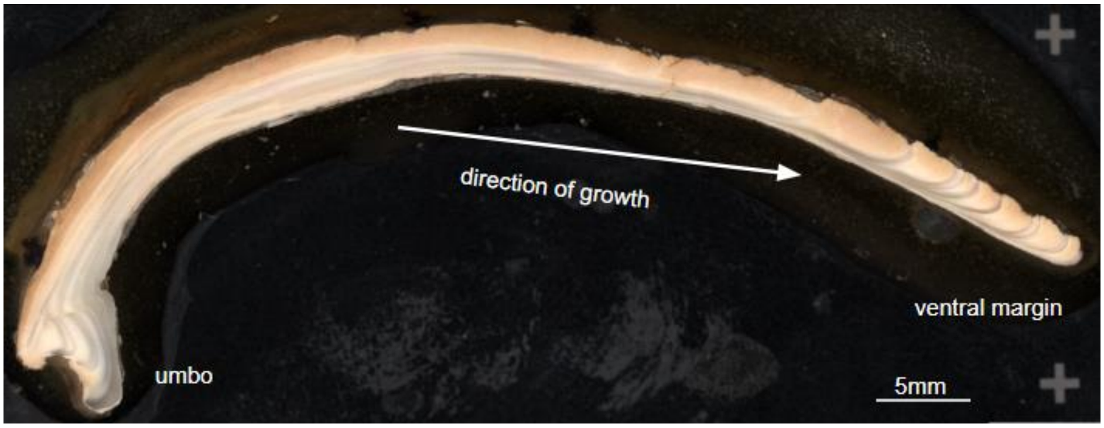

Figure 1.3 Thick section of *Mya arenaria* with annual growth bands visible at the chondrophore. The dotted line in a) indicates where the cross section was cut.

growth increments (Edge et al., 2022; Hadden et al., 2023; Jones et al., 2010; Sherwood et al., 2008). This is not only true for carbon isotopes. Studying the growth increments in the mollusk shell carbonate — a field of study known as sclerochronology — can be used to determine the ontogenetic age of the mollusk at the time of harvest and even the season of harvest by means of the oxygen isotope signatures which can be used to reconstruct past sea surface temperatures (Burchell et al., 2014, 2018; Hallmann et al., 2009).

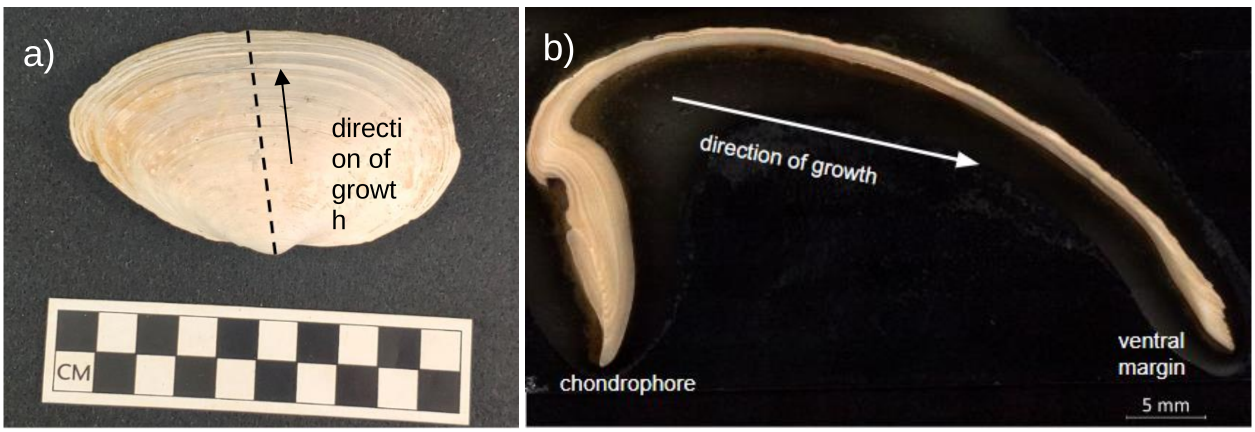

Figure 1.4 Thick section of *Saxidomus gigantea* showing a cross section of the shell with annual growth lines visible at the ventral margin.

Radiocarbon measurements from marine shells are also subject to time-averaging, which is when carbonate spanning many growth increments and potentially years of growth is sampled together. Effects of time averaging are highly dependent on sampling strategies and species-specific growth patterns of mollusks as the grouping of annual growth increments is not homogenous throughout most shells, for example consider the growth lines in *Mya arenaria* (Figure 1.3) and *Saxidomus gigantea* (Figure 1.4). Time averaging effects introduce a source of uncertainty that may be difficult to quantify and when radiocarbon data from marine shells are used with a contemporaneous terrestrial sample to calculate ΔR, this time averaging effect will also reduce the accuracy of the calculated ΔR value.

1.5 RADIOCARBON DATES AND MARINE SHELL: A FOCUS ON CANADIAN ARCHAEOLOGY

While there is a substantial knowledge base on marine radiocarbon data and calibration in the broader archaeological community, it is significantly lacking in Atlantic Canada. A quick search on the Canadian Archaeological Radiocarbon Database reveals the lack of radiocarbon data in Nova Scotia, and the wider Atlantic Canada, compared to other Canadian provinces (Martindale et al., 2016). As shown in Table 1, the Atlantic provinces — Prince Edward Island, Nova Scotia, and New Brunswick, excluding Newfoundland and Labrador — are the three provinces with the lowest number of reported dates on the CARD database.

Not only is there an overall shortage of radiocarbon data in Atlantic Canada, but there is also a lack of research focusing on marine radiocarbon. Compare this to British Columbia, where multiple archaeological values for ΔR have been reported all along the Pacific Coast of the province (Carlson et al., 2017; Edinborough et al., 2016; Martindale et al., 2018). Studies in British Columbia even include work on analyzing the variability of the reservoir ages over-time (Southon & Fedje, 2003) and suggestions for processing large amounts of ΔR values to build confidence in estimates (Martindale et al., 2018). No such bank of archaeological data exists for Atlantic Canada and very few archaeological ΔR values are reported for this region. In particular, only ΔR values available for Nova

Scotia on the online database (Reimer & Reimer, 2001) are from a 2006 GEOSCAN survey which reports only modern values for the region (McNeely et al., 2006).

Table 1.1 Assessment of reported radiocarbon dates on the CARD database for each Canadian province. Nova Scotia has the second lowest number of reported radiocarbon dates in the country.

Canadian Province	CARD Database	
	Number of Sites	Number of Samples
Alberta	336	1186
British Columbia	564	2344
Manitoba	119	337
New Brunswick	54	141
Newfoundland and Labrador	196	675
Nova Scotia	38	85
Northwest Territories	185	458
Nunavut	544	1407
Ontario	330	950
Prince Edward Island	8	23
Quebec	265	744
Saskatchewan	138	381
Yukon	175	572

In this thesis, I focus closely on Port Joli Harbour Nova Scotia. This harbour, located in south west Nova Scotia has the highest density of shell middens in Atlantic Canada and has been continuously occupied for at least the last 1500 years (Betts, 2019).

The fully terrestrial chronology for Port Joli Harbour is an exceptional resource for studies of marine radiocarbon in Atlantic Canada as it provides a point of comparison between terrestrial radiocarbon dates and radiocarbon dates from marine shell. The previous archaeological work in the harbour (see Betts, 2019; Betts et al., 2017; Burchell et al., 2014) provides one of the few archives in Atlantic Canada where so many shell samples are available and have the potential to be correlated with existing terrestrial dates.

Despite the abundance of radiocarbon dates and the abundance of coastal shell midden sites in Canada, discussions surrounding building confidence in these data is one that has only recently started to gain more traction among archaeologists (Hadden et al., 2023; Lotze et al., 2022). This discussion is also deeply intertwined with questions of intra-shell radiocarbon variability and the accuracy and precision of ΔR measurements: how to measure them, how to report them, how to use them, and their effect on archaeological chronologies.

1.6 THESIS STRUCTURE

This thesis is a manuscript-based body of work. Chapter 1 provides an overview of the necessary technical information regarding radiocarbon dating — including calibration and the marine reservoir correction —, mollusk shell growth strategies and shell structure, and the feminist approach to contextualize the two manuscripts. Chapter 2 is a draft of a manuscript co-authored by Dr. Kristin Poduska, Sarah Keuhn, Megan Mackinnon, and Dr. Meghan Burchell, to be submitted to the Journal of Archeological

Science Reports. Chapter 3 is a draft of a manuscript co-authored by Dr. Matthew Betts, Dr. Kristin Poduska, and Dr. Meghan Burchell.

1.6.1 Manuscript 1: Intra-shell Radiocarbon Variability In Marine Mollusks: Considerations for Archaeology and Shell Midden Chronologies

Intra-shell radiocarbon variability is affected by both geographic location and the growth strategies of the mollusk which dictates how the shell $CaCO_3$ is formed. We consider three bivalve mollusks species from three different coastal locations in Canada: *Saxidomus gigantea* from British Columbia, *Crassostrea virginica* from Prince Edward Island, and *Mya arenaria* from Nova Scotia. Each of these species exhibits different growth patterns, different mineralogies, and originates from different marine contexts resulting in different degrees of intra-shell ^{14}C variability. This manuscript outlines the importance of considering growth strategies and ontogenetic growth when deciding which species and which individual samples should be used for chronometric dating.

Co-authorship statement

In this chapter, all radiocarbon calibrations to the 2020 calibration curves were completed by me and I prepared samples, collected, and analyzed all FT-IR data from the marine shell samples. The shell samples in this chapter are from several previous excavations and were stored in the archives at Memorial University of Newfoundland. The excavations were completed as follows: Pitawelkek, Prince Edward Island excavated by Dr. Helen Kristmanson, excavations at Port Joli, Nova Scotia completed by Dr. Matthew Betts, and excavations at Deep Bay and Comox, British Columbia completed by Dr. Greg Monks. Preparation for imaging of samples was completed by Sarah Keuhn, Megan

Mackinnon, and me, and I designed all the figures. Dr. Burchell and I both designed the sampling strategies for the shell samples, I completed all data analysis and synthesis, and I am the primary author of this manuscript.

1.6.2 Manuscript 2: Refining the Radiocarbon Chronology at Port Joli Harbour, Nova Scotia, Canada with Marine Shell Dates and Local ΔR

The second manuscript focuses on Port Joli Harbour as a case study for investigating the effects of sampling strategies on ΔR calculations. This harbour, located on the southwest coast of Nova Scotia in the ancestral land of the Mi'kmaq, has over 20 shell middens throughout the perimeter of the harbour and has completely terrestrial chronology which reports continuous occupation throughout the last 2000 years (Betts, 2019). By analyzing ^{14}C from shell samples from the same stratigraphic context as previously dated terrestrial samples, the viability of *M. arenaria* shells for radiocarbon analysis is assessed and a fully marine chronology is constructed for the harbour which serves as a point of comparison to the previously published terrestrial chronology (Betts, 2019). Using these paired marine-terrestrial samples, we also take a preliminary look at calculating an archaeological ΔR value for the harbour and highlight the challenges in calculating this value from shell samples, but also the opportunities available to expand our understanding of human-environment interactions in the harbour by connecting local marine changes to wider climate trends (Lotze et al., 2022).

Co-authorship statement

All shell samples in this manuscript were excavated by Dr. Matthew Betts and were curated at the Canadian Museum of History prior to being analyzed at Memorial University. I selected all samples for radiocarbon analysis, pre-sectioned them and removed remineralized carbonate prior to shipping to the AMS lab at the University of Ottawa. I also collected all of the FT-IR data independently, calibrated all samples and calculated all ΔR values myself using the online tool (R. W. Reimer & Reimer, 2017). I am the primary author of the manuscript, and I designed all figures.

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CHAPTER 2

INTRA-SHELL RADIOCARBON VARIABILITY ACROSS CANADA: CONSIDERATIONS FOR CANADIAN COASTAL ARCHAEOLOGY AND SHELL MIDDEN CHRONOLOGIES

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ABSTRACT

Shell middens are a valuable archive for high-resolution climate studies in archaeological contexts and are valuable for radiocarbon analyses because of their abundance at midden sites in comparison with other materials. However, variability in radiocarbon measurements from marine mollusks poses a challenge for obtaining secure chronometric dates for archaeological applications. We compare intra-shell radiocarbon variability from three different mollusk species from three different archaeological contexts from across Canada: *Saxidomus gigantea* from British Columbia, *Mya arenaria* from Nova Scotia, and *Crassostrea virginica* from Prince Edward Island. Growth increment analysis of the mollusk's shells is useful for understanding time-averaging effects in the radiocarbon measurements. Obtaining multiple samples per shell and conducting a chi-squared test can help determine when intra-shell radiocarbon variability is significant. Shorter lived *C. virginica* shells displayed the least degree of intra-shell variability. Both *M. arenaria* from Nova Scotia and *S. gigantea* from British Columbia displayed significant differences in their intra-shell measurements. The growth strategies of *C. virginica* make this species less susceptible to adverse time-averaging effects in intra-shell measurements, but

sclerochronological analyses should still be completed to ensure short lived samples are selected.

2.1 INTRODUCTION

In coastal archaeology sites, specifically shell middens, marine mollusk shells are found in abundance and these materials can provide a high-resolution archive of past oceans, people, and time. The $CaCO_3$, of which most mollusk shells are composed - along with chitin (Marin et al., 2012) - combined with their good preservation in archaeological contexts makes them a common choice for radiocarbon dating shell midden sites and for calculating local marine reservoir value (ΔR). However, intra-shell variability complicates this process. Marine mollusks biomineralize their shells in equilibrium with the environment in which they live and thus the radiocarbon signature in their $CaCO_3$ shells closely reflects the radiocarbon signature in the seawater. While the contribution of respired metabolic to the ^{14}C signatures of mollusk shells will vary between species, it is thought that this number remains below 10%, and variations in marine shell radiocarbon are mainly attributed to variations in the local marine environment due to factors such as upwelling and freshwater input (Culleton et al. 2006, Hadden & Cherkinsky 2017, Hadden et al., 2023). As shells grow and subsequent layers of $CaCO_3$ are deposited, the radiocarbon signature of the seawater may change, resulting in changes in the radiocarbon signature of the shell.

Although intra-shell radiocarbon variability is not a new phenomenon, it is often a more prominent focus in the fields of palaeoclimatology and geological science contexts -

where intra-shell measurements may be used to construct chronologies of marine reservoir changes over time (see Butler et al., 2009; Edge et al., 2022; Lower-Spies et al., 2020) - rather than in archaeological contexts, and discussions of this phenomenon in Canadian archaeology are severely lacking. There is growing overlap between palaeoclimate, geological sciences, and archaeology, and the uncertainties associated with radiocarbon data that stem from sampling strategies and ΔR corrections are starting to be communicated more effectively in archaeological literature (see Hadden et al. 2023). However, in archaeological applications when marine shell radiocarbon is used as a chronometric age measurement, questions of variability are still frequently addressed inadequately. Considering the sources of variability in radiocarbon dates from marine shell can inform sampling strategies and build confidence in marine shell dates for archaeological applications (Culleton et al. 2006, Hadden & Cherkinsky 2017).

Here we investigate intra-shell radiocarbon variability in three different mollusk species from archaeological contexts in three different coastal Canadian provinces: *Saxidomus gigantea* from British Columbia, *Mya arenaria* from Nova Scotia, and *Crassostrea virginica* from Prince Edward Island. We highlight the interplay between mollusk growth strategies and sampling strategies and highlight the stack-up of uncertainties due to these factors and ΔR values in the final calibrated values. By analyzing the uncertainties on the uncalibrated radiocarbon ages and the ΔR values available in the literature, we highlight how the interplay between different growth strategies and sampling strategies will affect the measured variability. We also make suggestions for analyzing uncertainties in order to build confidence in the use of marine shell radiocarbon for one-off chronometric dates and select the best samples for analysis even in scenarios where there are no terrestrial samples to compare the marine measurements to. Intra-shell radiocarbon variability in these Canadian coastal regions have not been studied prior to this work and we hope that this study will encourage Canadian archaeologists to incorporate these considerations into their sampling and analysis decisions.

2.1.1 Marine Shells and Radiocarbon

The challenges surrounding radiocarbon dating marine shells have long been known. Douka et al. (2010) provide a detailed outline of many of the challenges involved in radiocarbon dating marine shell, including: local and global marine reservoir effects, the hardwater effect, the old-shell problem, and recrystallization. These effects all contribute to additional errors on radiocarbon measurements other than those resulting from the accelerated mass spectrometry measurement (AMS) process and thus, are difficult to quantify. Some of these challenges are easier to address than others, for example detecting recrystallization using techniques like Fourier Transform Infrared Spectroscopy (FT-IR), Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD) is relatively straightforward. But selecting a local marine offset (ΔR) with a high degree of spatial and temporal accuracy for the sample in question is often challenging. Understanding how much the choice of ΔR value and the shell sampling strategies affect the calibrated age ranges used for archaeological interpretation is difficult to characterize and is often not addressed in archaeological contexts.

In the North Atlantic, samples with known collection years are used for building large chronologies of marine ^{14}C over time and cross-dating shell growth increments. The species *Arctica islandica* has been used widely for these purposes (Butler et al., 2009; J. Scourse et al., 2006; J. D. Scourse et al., 2012). In situations where long-lived species are used to build large chronologies, the ^{14}C measurements are not used to date the mollusk samples themselves, as they have already been fit into a chronology and an absolute date can be determined. Rather, the ^{14}C measurements are used to analyze temporal variations in marine upwelling and construct chronologies for ΔR. Similarly, in South America, marine radiocarbon variation from archaeological mollusk samples has been used to study El Niño events on the Pacific coast (Andrus et al., 2005; Etayo-Cadavid et al., 2013). Intra-shell measurements from shorter-lived species --- such as *Mytilus californianus* and *Crassostrea virginica* --- with a known date of death have also been used to study changes in ΔR over time in California and Florida (Culleton et al. 2006, Hadden & Cherkinsky, 2017, Rick et al. 2012).

The studies described above rely on either a known collection date, a contemporaneous terrestrial radiocarbon date, or an existing chronology to anchor the marine radiocarbon measurement in time and use radiocarbon measurements as palaeoclimate data rather than a chronometric age measurement. Even if marine shell samples are to be sampled so as to increase time-averaging effects to average over any ^{14}C variability, the intra-shell radiocarbon variability should still be considered to understand its effects on the final value used for one-off chronometric dating (Hadden & Cherkinsky, 2017, Hadden et al., 2017).

2.1.2 Local and Global Marine Carbon Reservoirs

The ocean acts as a carbon reservoir, locking old carbon in the deep seawater in the form of dissolved inorganic carbon (DIC) for lengths of time much longer than that of the half-life of radiocarbon. This carbon locking results in the isotopic signature of the global carbon signature of the seawater having a much older apparent radiocarbon age than contemporaneous terrestrial samples. This offset between the global ocean carbon reservoir and the global atmospheric carbon reservoir is accounted for by the construction of different calibration curves - marine and terrestrial (Heaton et al., 2020; P. J. Reimer et al., 2020). However, local ocean upwelling and mixing patterns, input of freshwater runoff or other inputs of old carbon such as limestone leaching will further affect this offset both spatially and temporally and can result in older or younger apparent marine ages (Douka et al., 2010; Hadden et al., 2023). For mollusks living in coastal zones, the combination of global and local carbon reservoir offsets will be reflected in the shell's chemistry and will result in older apparent ages than those of contemporaneous terrestrial samples.

The local marine reservoir offset is an important value for both correctly calibrating marine dates and for determining oceanographic and climate conditions. When calibrating radiocarbon dates from any marine sample, the ΔR value is a correction that is applied to the measurement prior to calibrating to the appropriate marine curve. The most recent marine calibration curve is the Marine20 curve (Heaton et al., 2020), which can be interpreted as a global average of the marine reservoir, is constructed using tropical and subtropical records from cross-dated corals and serves as a starting point for calibration, but which requires corrections for regional variations (P. J. Reimer et al., 2013). It should be noted that regional ΔR values may not be static and will change in time with variations in ocean circulation, freshwater runoff, and larger climatic events.

2.1.3 Sources of Radiocarbon Variability in Shells

Molluscan growth patterns may differ from species to species, and even from individual to individual. Thus, intra-shell ^{14}C profiles are dependent on the growth strategy of the specific mollusk and may further be affected by post-depositional changes to the isotopic chemistry (diagenesis). Measured intra-shell radiocarbon variability is a combination of three different effects: intrinsic variability which exists in pristine shells, variability resulting from diagenesis, and fabricated variability which is the result of sampling strategies.

Intrinsic Variability

Intrinsic variability is termed here as such because it represents actual variability in the ^{14}C content of the shell due to the interplay between variation in the ^{14}C in the environment and the growth patterns of the shell. Changes in the local reservoir (ΔR) during the mollusk's lifetime will be reflected in the profile of the ^{14}C content throughout the shell carbonate (Andrus et al., 2005; Culleton et al., 2006; Etayo-Cadavid et al., 2013; Hadden & Cherkinsky, 2017; Jones et al., 2010; Jones et al., 2010). This intrinsic variability is also the product of the growth patterns of the shell as shells rarely tend to grow linearly, rather they usually deposit carbonate at different rates during different times of the year, producing a ^{14}C profile that may not match that of the environment directly (Burchell et al., 2014; Cannon & Burchell, 2009). The contribution of respired carbon to the mollusk's ^{14}C signature can also be categorized as intrinsic variability although it is assumed to be minimal (Hadden et al., 2023; Lindauer et al., 2022).

Diagenetic Variability

It is standard practice that marine shell samples are screened for any diagenetic alterations to the carbonate material, and the diagenetically altered shell material is removed before the material is radiocarbon dated (Crann et al., 2017). For some species, changes in the ^{14}C content of the shell are analyzed indirectly by characterizing changes in the mineralogy of the shell. For example, for shells whose carbonate is primarily aragonite, a common form of diagenesis for aragonitic shells is the dissolution and reprecipitation of the aragonite as calcite - which has a different crystal structure. The dissolution and reprecipitation process can incorporate either younger carbon or "dead" carbon into the shell material and thus may skew the radiocarbon age, making it less representative of the actual age of death of the mollusk. Some methods that can be used for determining the shell's mineralogy are Fourier Transform Infrared Spectroscopy (FT-IR), X-ray diffraction (XRD), Raman spectroscopy, and Feigl's solution. Identifying effects of diagenesis is more challenging for mollusk species that have mixed calcite-aragonite mineralogies, like oysters, because the simple detection of calcite does not necessarily indicate diagenesis (Bowman 1990).

Fabricated Variability

The reported variability in intra-shell radiocarbon measurements is highly dependent on sampling strategies. If portions of the shell are averaged by radiocarbon dating larger sections of shell, then the dates returned will be time-averaged potentially across years of growth. Precisely drilled or micro milled samples are less susceptible to time averaging but are more sensitive to capturing possibly anomalous upwelling or freshwater influxes and thus dramatic changes in the marine environment (Culleton et al. 2006). These more precisely drilled samples are also limited by the required sample size for radiocarbon measurements, and smaller samples have a larger risk of loss or damage during the sample preparation process. For this reason, radiocarbon measurements may be taken on a larger area of shell (on the order of several millimeters) to ensure no sample loss or damage, and to allow for the roughly 20% reduction in mass of the sample due to the acid etching (Crann et al., 2017; Culleton et al., 2006; Hadden & Cherkinsky, 2017; Helama & Hood, 2011).

2.2 STUDY AREAS AND SAMPLES

To highlight the importance of considering each geographic region and each species on a case-by-case basis, we have chosen three different mollusk species with archaeological significance from three different coastal regions in Canada: *Saxidomus gigantea* from British Columbia, *Crassostrea virginica* from Prince Edward Island, and *Mya arenaria* from Nova Scotia.

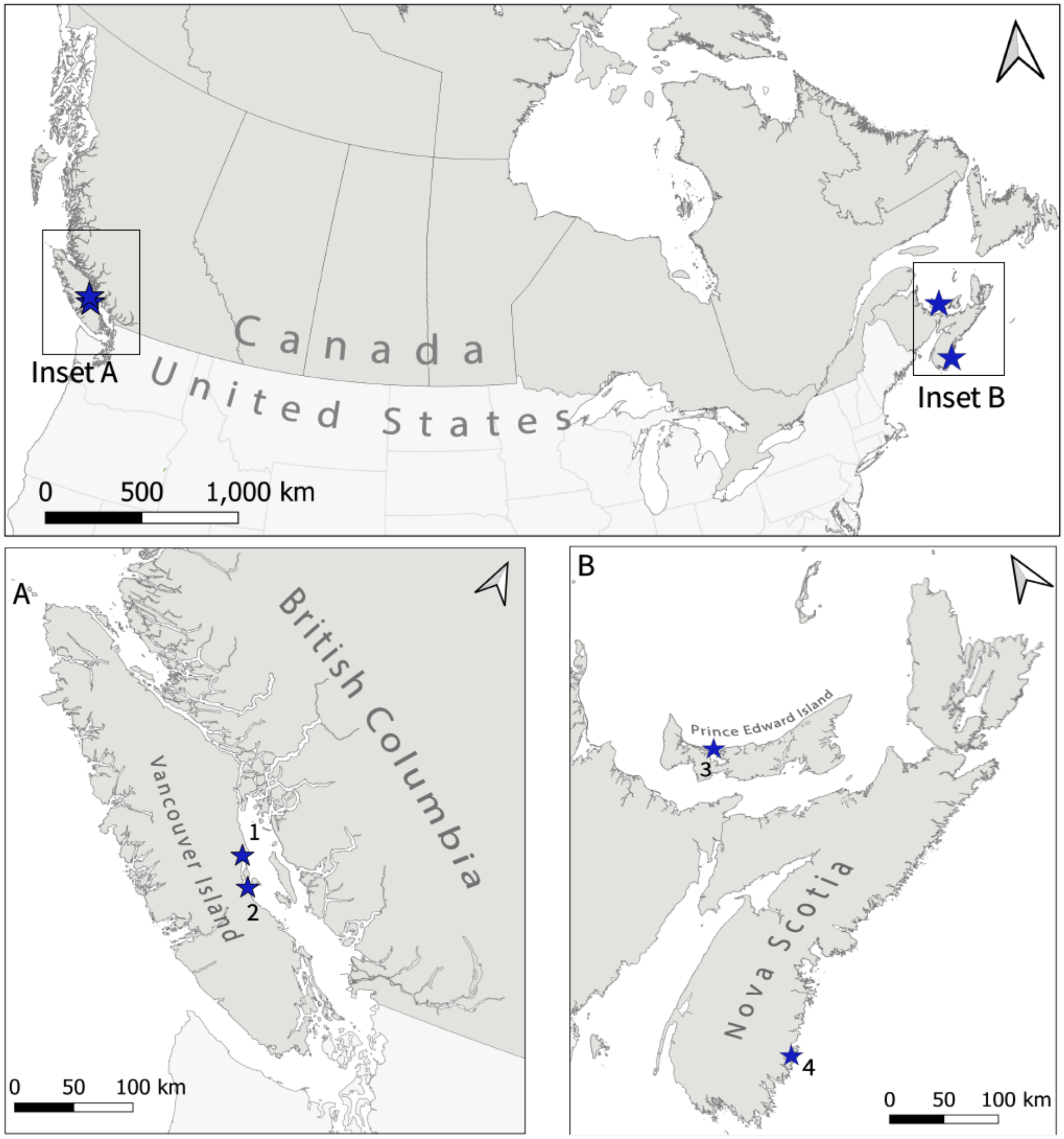

Figure 2.1 Map of Canada showing archaeological midden sites from which our samples originate. The numerical labels designate the sites as follows: 1) site DkSf-20, located in modern day Comox, British Columbia; 2) site DiSe-7, located in Deep Bay, British Columbia; 3) Pitawelkek (site CdCw-5), located on Hog Island, Prince Edward Island; and 4) site AlDf-24, located in Port joli Harbour, Nova Scotia.

2.2.1 Deep Bay and Comox, British Columbia: *Saxidomus gigantea*

British Columbia has been a hotspot in Canadian archaeology. Sites such as Namu and Pender Island have become common in Canadian coastal archaeology discourse (Burchell et al., 2013; Carlson et al., 2017; McLaren et al., 2015). The samples analyzed in this paper were excavated from Deep Bay, DiSe-7 in the late 1970s (Monks, 1977) and Comox (K'omoks) British Columbia in 2017 during the Simon Fraser University Archaeological Field School. Both Comox and Deep Bay are located on the east coast of present-day Vancouver Island along the Salish Sea, in the traditional territory of the Coast Salish and Shishalh peoples and are labeled as 1 and 2 respectively in Figure 2.1. The archaeological records in the Salish Sea span the length of the Holocene (Greene et al., 2015; Moss et al., 2007).

Saxidomus gigantea, also called the butterclam, is a marine mollusk found in intertidal zones along the west coast of North America extending all the way from California to Alaska (Hiebert, 2015). The shell of this species is composed entirely of aragonite with an inner nacreous aragonite layer and the outer layer, which contains readable growth increments, being crossed-lamellar aragonite (Gillikin et al. 2013). The samples used in this work are from the Salish Sea in British Columbia, a region with rich archaeological history spanning the length of the Holocene. *S. gigantea* has been harvested by coastal populations throughout the Holocene in this region and has been used by archaeologists to determine season of harvest and to reconstruct past sea surface temperatures in the Salish Sea (Burchell et al., 2013; N. Hallmann et al., 2013; Nadine Hallmann et al., 2009)**.** The lifespan of *S. gigantea* is 20+ years (Hiebert, 2015). As *S.*

gigantea reaches senility, the growth lines at the ventral margin of the shell become more compact and cluster together, while growth lines in the middle of the shell may be more spread out. The umbo of *S. gigantea* contains a complete record of ontogenetic growth but the carbonate in this region is skewed toward younger growth. The umbo is not used for growth increment analysis to determine ontogenetic age as older growth lines cluster together and are not distinguishable.

2.2.2 Port Joli Harbour, Nova Scotia: *Mya arenaria*

Located on the east coast of Canada, Nova Scotia is the traditional territory of the M'ikmaq people. Port Joli Harbour is an inlet in Southwestern Nova Scotia that contains the largest density of shell middens in Atlantic Canada. Samples in this study were collected from Port Joli Harbour during the E'se'get archaeological project in 2009. This extensive, community-based research project led by Dr. Matt Betts from the Canadian Museum of History in Ottawa, resulted in the publication of a comprehensive overview of the archaeological history of the harbour (Betts, 2019; Hrynick et al., 2017; Hrynick & Betts, 2017). Site AlDf-24 in Port Joli Harbour is labeled as 4 in Figure 2.1.

Mya arenaria, the soft-shelled clam, has a large ecological range, extending along both the Pacific and Atlantic coasts of North America and the Atlantic coast of Europe as well as low- salinity areas in Europe (Heibert, 2015). The shell of *M. arenaria* is also composed entirely of aragonite. The samples studied in this work are from Port Joli, Nova Scotia, a Mi'kmaq archaeological site that has been inhabited continuously for at least the last 1500 years. Similarly, to *S. gigantea*, *M. arenaria* has been harvested by coastal populations for food and constitute much of the material from the shell middens in Port Joli. Unlike *S. gigantea*, *M. arenaria* has a chondrophore: a protruding piece of shell at the hinge that is quite robust and preserves well in the archaeological record. The chondrophore also contains a complete record of growth, with all the annual growth lines being visible. *M. arenaria* lives up to about 20 years, although the recorded lifespans vary geographically (Heibert, 2015).

2.2.3 Pitawelkek, Prince Edward Island: *Crassostrea virginica*

Prince Edward Island is a small island province in Atlantic Canada. The midden site of Pitawelkek (CdCw-5, labeled as 3 in Figure 2.1), from which the samples in this paper were obtained, has evidence of continuous occupation extending back to about 700 AD based on terrestrial AMS dates, but diagnostic artifacts found at the site suggest earlier occupations extending back about 2000 years from the present (Kristmanson, 2019). Pitawelkek is located on Hog Island, North-East of Malpeque Bay and the site holds lasting significance to the Mi’kmaq people of Prince Edward Island (Kristmanson, 2019).

Crassostrea virginica, also known as the Eastern oyster, can live up to 20 years and is a fairly robust species, being able to withstand low temperatures (Herbert & Steponaitis, 1998). The mineralogy of the oyster is more complex than the other two species considered here as it consists of both calcite and aragonite (Carriker & Palmer, 1979; Marin et al., 2012). Particularly, the hinge region of the shell of *C. virginica* contains alternating layers of calcite and aragonite (Carriker & Palmer, 1979). Similar to

M. arenaria, *C. virginica also* contains a record of all annual growth lines at the hinge and is the portion of the shell that is most robust for growth increment analysis (Andrus & Crowe, 2000; Herbert & Steponaitis, 1998; Zimmt et al., 2019).

2.3 METHODS

Our analysis methods involve two key components. Accelerator mass spectrometry (AMS) radiocarbon analysis was performed at the Lalonde AMS laboratory at the University of Ottawa to radiocarbon date multiple samples per shell. Growth line analysis was used to determine an estimate of the ontogenetic age of the mollusk, as well as how many years of growth were captured in each sample submitted for radiocarbon dating.

2.3.1 Radiocarbon Dating

To ensure no time-averaged carbonate was captured in the radiocarbon analysis, the innermost nacreous layer of *S. gigantea* samples was removed with a Dremel to ensure no time-averaged carbonate material was captured in the radiocarbon analysis. At the ventral margin of *M. arenaria* samples, the inner nacreous layer was removed where it could visually be detected. This layer was not present on *C. virginica* samples. Additionally, samples were analyzed using Fourier Transform Infrared Spectroscopy (FTIR) to ensure that their mineralogies matched that expected for their respective species. More details can be found in Appendix A.1.

To analyze intra-shell radiocarbon variability in *S. gigantea*, three shells were selected and sampled at three different locations on the shell: at the umbo, in the middle, and at the ventral margin. For *M. arenaria*, the shell was sampled at two locations: at the chondrophore and at the ventral margin, and for *C. virginica* the shells were also sampled at two locations: the hinge and the ventral margin. Sampling locations are shown in Figure 2.3. Figure 2.3 shows the size of the sampling locations for radiocarbon analysis on each shell sample.

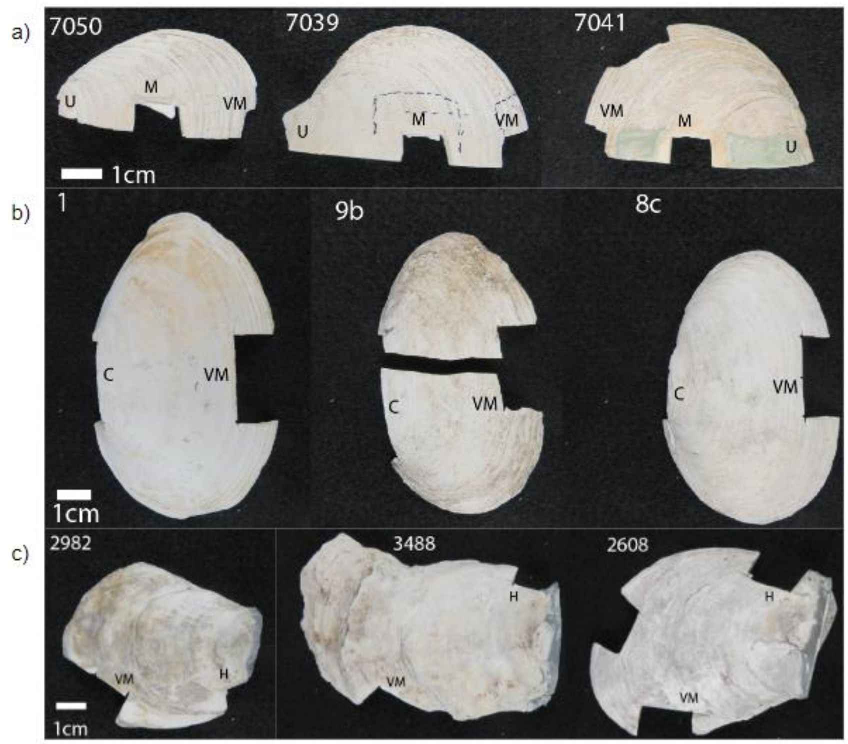

Figure 2.2 Sampling locations for radiocarbon analysis for a) *S. gigantea* b) *M. arenaria* and c) *C. virginica*. *S. gigantea* was sampled at the umbo (U), middle (M), and ventral margin (VM), *M. arenaria* was sampled at the chondrophore (C), and ventral margin (VM), and *C. virginica* was sampled at the hinge (H) and ventral margin (VM).

Shell samples were prepared and analyzed for radiocarbon at the Lalonde AMS laboratory from the University of Ottawa in July of 2018. Following the procedures outlined in (Crann et al., 2017), all samples were pre-etched in HCl to remove the outermost 20% of shell, ensuring that any surface contaminants are removed. To isolate the carbon from the carbonate samples, the remaining shell material was crushed with a mortar and pestle and reacted with phosphoric acid (H_3PO_4) which produces carbon dioxide (CO_2), water (H_2O), and calcium phosphate ($Ca_3(PO_4)_2$). The resultant CO_2 was collected and subsequently graphitized in the presence of iron and hydrogen to produce elemental carbon which was then analyzed by the accelerator mass spectrometer system. Samples were corrected for fractionation by normalizing the $^{13}C/^{12}C$ ratios to -25 with respect to the Pee Dee Belemnite standard. Values are reported as fractionation corrected uncalibrated years before present (BP).

2.3.2 Imaging

Growth lines were analyzed to determine how many years of ontogenetic growth are averaged over in each of the radiocarbon measurements. For *S. gigantea* samples the side from which samples were cut for radiocarbon measurements were cut down to produce a flat cross section with the portions that were analyzed for radiocarbon being marked on the surface of the shell with a marker. The cross section of the shell was sealed to a glass slide with Hillquist epoxy resin and cut using a Buehler IsoMet 1000 precision saw to create a thick section (3mm). The *M. arenaria* chondrophores were similarly prepared as thick sections. For the hinge and ventral margin portions of *C. virginica* and ventral margin portions of *M. arenaria* samples, additional portions of the shell were cut immediately adjacent to the portions used for radiocarbon analysis for imaging. *C. virginica* samples were prepared with Struers epoxy resin and *M. arenaria* samples were prepared with Buehler's epoxy resin, with the cross section of each portion flush with the bottom of the puck.

The samples were ground and polished to produce an optically smooth surface for imaging using a Buehler MetaServ 250 grinder-polisher and glass plates with 800 and 1200 grit size Silicon Carbide grinding paper until optically flat. For the *S. gigantea* thick sections, the segments that were sampled for radiocarbon were marked on the exterior of the shell with a marker and are indicated by the white boxes in the figures. All images were taken on the Zeiss Axio Zoom.V16 microscope.

2.4. RESULTS

First, the results of FT-IR analysis are shown to determine if samples are calcite, aragonite, or a mixture of the two. Samples of *M. arenaria* and *S. gigantea* are aragonite and *C. virginica* is a mixture: primary calcite with traces of aragonite. We then report the results of radiocarbon analysis, which display different degrees of intra-shell variability within samples of the same species and between species.

Microscope images of the cross sections of each sample submitted for radiocarbon analysis are first presented. These images were used to obtain an estimate of the years of ontogenetic growth captured in each sample. This data is then summarized with the radiocarbon results in section 2.4.2.

2.4.1 Imaging

The images of *S. gigantea*, *M. arenaria*, and *C. virginica* are shown in Figures 2.3, 2.4, and 2.5 respectively. In Figure 2.3, the regions which were sampled for radiocarbon are marked by white boxes. The samples displayed different degrees of readability, for example the ventral margins of *C. virginica* were challenging to interpret as these portions are typically not used for growth line analysis (Andrus & Crowe, 2000; Zimmt et al., 2019). These images were used to estimate the years of ontogenetic growth in each portion. These estimates are recorded in Table 2.1. Note that the inner nacreous layer on *S. gigantea* samples, which contains time averaged aragonite and no discernable growth increments, was removed to the best of our ability with a Dremel tool prior to radiocarbon analysis.

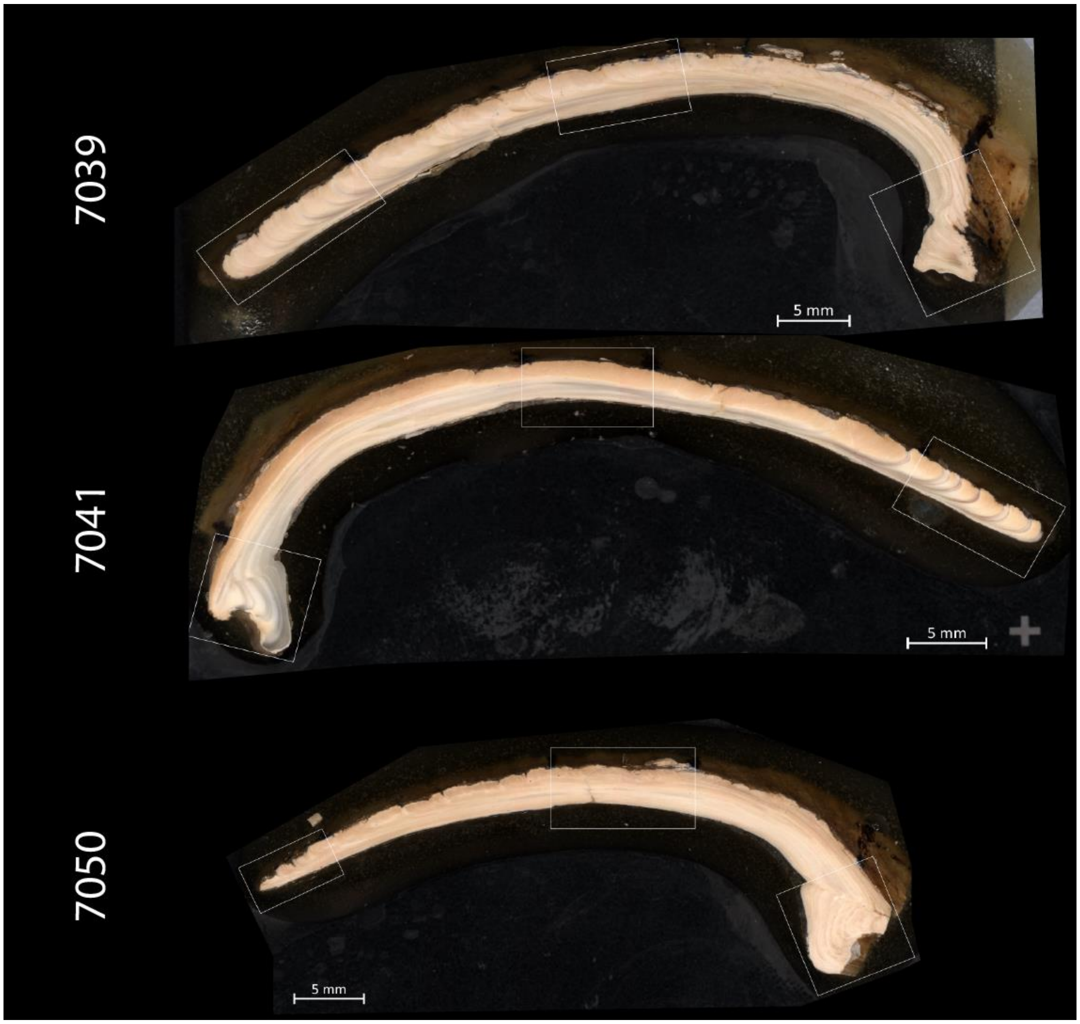

Figure 2.3 Thick sections of *S. gigantea* shells sampled for radiocarbon. For each shell, the boxes indicate the portions of the shell that were sampled for radiocarbon. For the *S. gigantea* samples, the inner remineralized layer which appears as grey in the figure was removed prior to radiocarbon analysis. Images were collected at 16X magnification.

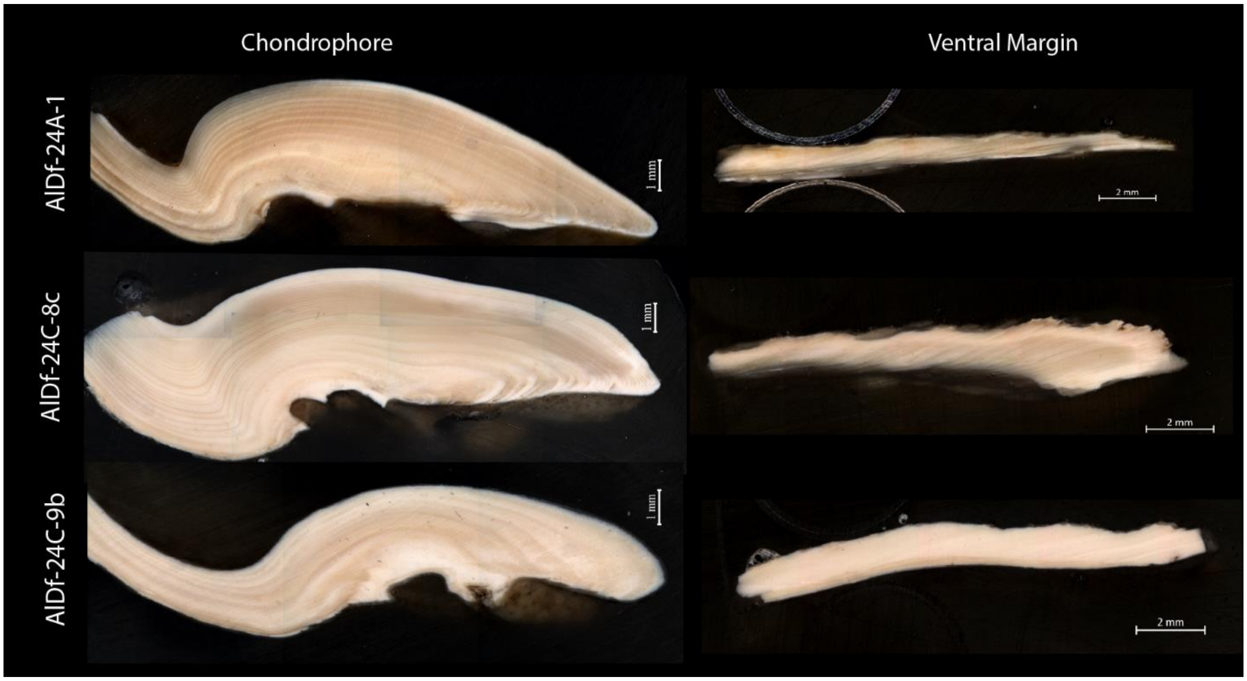

Figure 2.4 Thick sections of *M. arenaria* chondrophores and cross sections of ventral margins in epoxy from the same shell. Images were collected at 32X magnification.

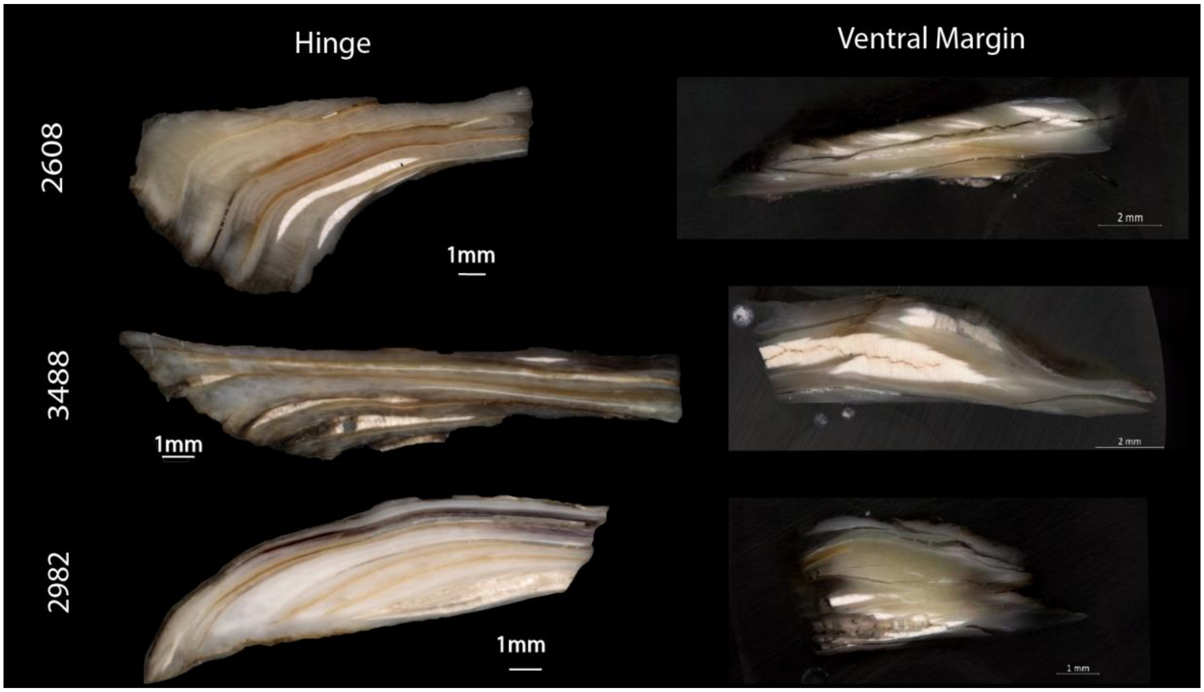

Figure 2.5 Thick sections in epoxy of the hinge and ventral margin portions of the *C. virginica* samples analyzed for radiocarbon. Ventral margin images were collected at 32X magnification and hinge sections were collected at 8.2X magnification.

2.4.2 Radiocarbon Dating

In Table 2.1 ontogenetic age estimates for each of the samples including an overall age estimate as well as an estimate for how much growth is incorporated into each of the sampled sections are recorded. The uncalibrated ^{14}C yr BP measurements are reported in Table 2.1 and are presented in Figure 2.6 to aid in the visualization of the error overlap. The R_Combine function in Oxcal was used to complete a chi-squared test following the procedure outlined in Ward and Wilson (1978). The R_Combine function returns the calculated T value, indicates whether it passes or fails at 5%, and reports a calibrated range for the combined dates.

The dates were calibrated using the Oxcal program (Heaton et al. 2020, Bronk Ramsey 2009). Calibrated dates are reported in calibrated years before present (cal BP) to the Marine 20 curve to 2σ (95.4% probability range). The ΔR values for Prince Edward Island, Nova Scotia, and British Columbia were taken from the online ΔR database and are updated to the Marine 2020 curve (Reimer & Reimer 2001).

All the oyster samples from P.E.I. have overlapping ^{14}C yr BP measurements and all shells pass the chi-squared test. One of the three *M. arenaria* samples does show a difference in ^{14}C yr BP measurements within the uncertainties reported (sample 9b) and a failing T value. Growth line analysis indicates that this mollusk is only 6 ontogenetic years old, this relatively short time suggests that the reported variability is not only the result of time-averaging but may be due to changes in the ΔR over the mollusk's life.

Table 2.1: Radiocarbon data obtained from *C. virginica* shells from Malpeque Bay, P.E.I., *M. arenaria* shells from Port Joli, N.S., and *S. gigantea* shells from Deep Bay and Comox, B.C. The ΔR values were retrieved from the online database (Reimer & Reimer 2001).
[1]ΔR=-77±77; weighted mean of 10 closest values for Malpeque Bay, PEI
[2]ΔR=-88±45; weighted mean of 10 closest values for Port Joli, NS
[3]ΔR=194±95; weighted mean of 10 closest values for Comox, BC

Location	Site	Lab ID	OtherID	Mineralogy	Species	Sampling location	Ontogenetic age (years)	Approximate age (years)	14C age (BP, uncalibrated)	Error	T value, pass/fail	Cal BP (Marine20, 2σ)
Malpeque Bay, P.E.I.	CdCw-5	UOC-18205	2608	Calcite + trace aragonite	*C. virginica*	Ventral margin	4	~4	1107	11	T=0, pass	770-435[1]
		UOC-18204				Hinge		~4	1108	11		
Malpeque Bay, P.E.I.	CdCw-5	UOC-18207	2982	Calcite + trace aragonite	*C. virginica*	Ventral margin	4	~2	903	11	T=0.4, pass	612-255[1]
		UOC-18206				Hinge		~4	913	11		
Malpeque Bay, P.E.I.	CdCw-5	UOC-18209	3488	Calcite + trace aragonite	*C. virginica*	Ventral margin	3	~2	859	11	T=0.8, pass	558-184[1]
		UOC-18208				Hinge		~4	873	11		
Port Joli, NS	AlDf-24A	UOC-18193	1	Aragonite	*M. arenaria*	Ventral margin	~10	~2	1947	11	T=1.3, pass	1598-1290[2]
		UOC-18192				Chondrophore		~10	1965	11		
Port Joli, NS	AlDf-24C	UOC-18199	9b	Aragonite	*M. arenaria*	Ventral margin	~6	<1	1684	11	T=5.967, **fail**	-
		UOC-18198				Chondrophore		~6	1722	11		
Port Joli, NS	AlDf-24C	UOC-18202	8c	Aragonite	*M. arenaria*	Ventral margin	>15	~1	1352	11	T=0.1, pass	965-671[2]
		UOC-18201				Chondrophore		>15	1358	11		
Comox, BC	DkSf-20	UOC-7050	7050	Aragonite	*S. gigantea*	Ventral margin	~10	~4	2217	23	T=0.5, pass	1680-1199[3]
		UOC-8000				Middle		~3	2200	21		
		UOC-8001				Umbo		<1	2220	22		
Deep Bay, BC	DiSe-7	UOC-7039	7039	Aragonite	*S. gigantea*	Ventral margin	~20	~9	1622	23	T=8.367, **fail**	-
		UOC-7062				Middle		~1	1673	23		
		UOC-7063				Umbo		<1	1716	23		
Deep Bay, BC	DiSe-7	UOC-7041	7041	Aragonite	*S. gigantea*	Ventral margin	~12	~7	1290	23	T=0.7, pass	716-317[3]
		UOC-7998				Middle		<1	1301	21		
		UOC-7999				Umbo		<1	1315	21		

Sample 7039 of *S. gigantea* from British Columbia also returns a failing T value. This is the oldest *S. gigantea* sample and has a difference of up to 10 years of growth between the umbo and ventral margin with the ventral margin containing the last 9 years of growth and the umbo containing only the first year.

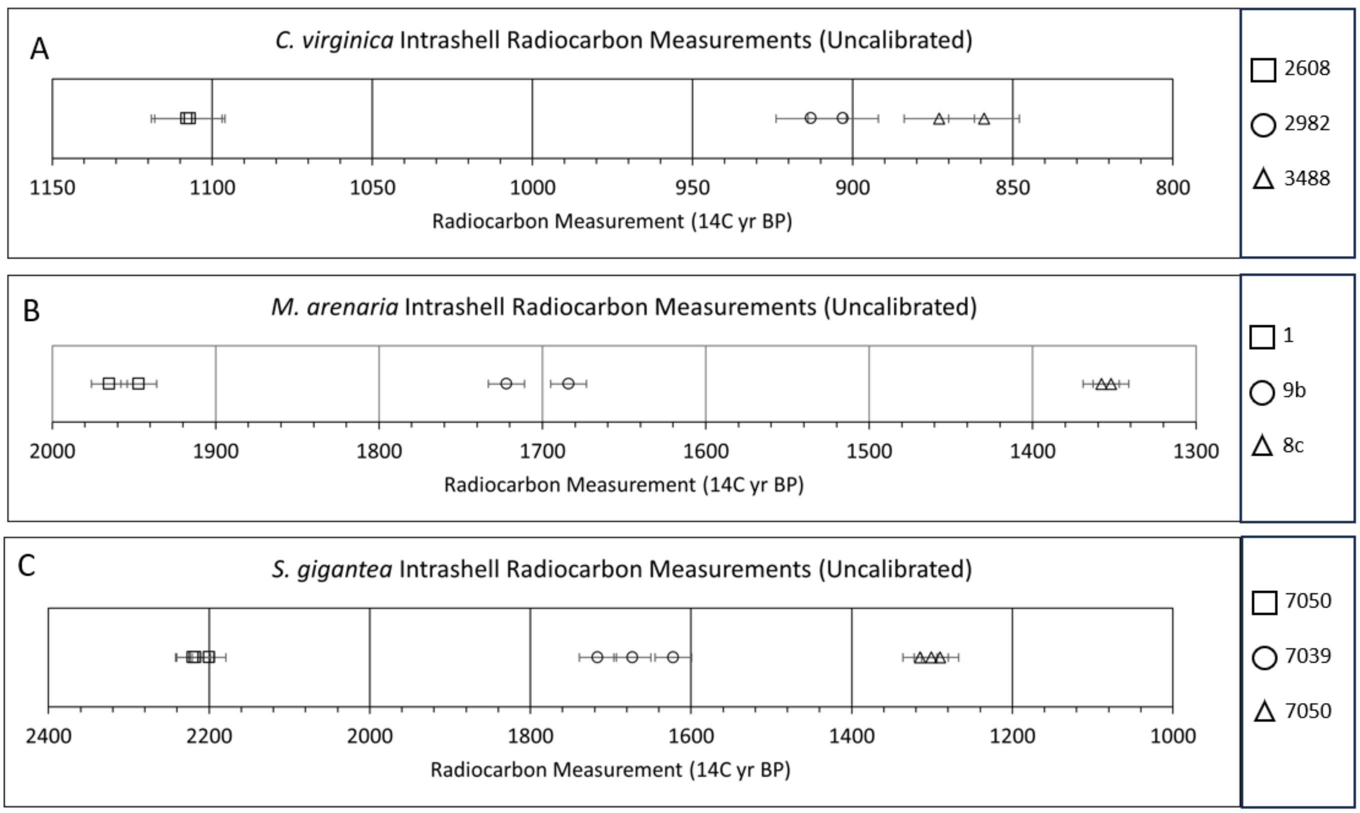

Figure 2.6: Uncalibrated intra-shell radiocarbon measurements from A) *C. virginica*, B) *M. arenaria*, and C) *S. gigantea*. The data are reported as 14C yr BP. The intra-shell measurements from *C. virginica* overlap within their uncertainties for all three specimens. Intra-shell measurements form *M. arenaria* sample 9b, and *S. gigantea* sample 7039 do not overlap within their uncertainties.

2.5 DISCUSSION

As discussed in previous sections, there are three main sources of variability when studying intra-shell radiocarbon variability: intrinsic variability, diagenetic variability, and fabricated variability. We have attempted to limit the contribution of diagenetic variability by using FT-IR to ensure that the samples of *M. arenaria*, *S. gigantea*, and *C. virginica* match their expected mineralogies. This data is presented in Appendix A. However, the contributions from intrinsic and fabricated variability to the radiocarbon measurements are intertwined. Other studies have reported intra-shell variability in radiocarbon measurements. For example, Jones et al. (2010) record an exceptionally large variation of 530 calBP within a shell from a *Mesodesma donacium* determined to be only ~5 yr in ontogenetic age. This variability is attributed primarily to changes in the local marine reservoir, suggesting that the total intra-shell ^{14}C variation can be interpreted as the minimum variation in the marine reservoir experienced throughout its life.

British Columbia is a coastal region with prominent seasonal upwelling variability, resulting in highly variable ΔR values due to the influence of the California current (Hadden et al. 2023). This makes this region, overall, more challenging to obtain secure radiocarbon dates, even with the use of short-lived species. This appears to also be the case for Nova Scotia. Nova Scotia is also located in a region of coastal variability that is subject to ocean-atmospheric effects like the North Atlantic Oscillation (NAO) (Lotze et al. 2022). The NAO operates on a multidecadal time scale and impacts precipitation, temperature, and coastal circulation patterns (Greene & Pershing, 2003; Lotze et al., 2022). Comparatively, P.E.I. is situated with the Gulf of St. Lawrence to the North and the Northumberland Strait to the south, and while P.E.I. is not subject to these major coastal current systems as British Columbia and Nova Scotia are, the ocean dynamics withing the Gulf of St. Lawrence are still complex (Galbraith et al., 2022). Studies focusing on ΔR calculation over space and time in this region are needed.

The *C. virginica* samples analyzed in this work produced robust radiocarbon dates, likely because they were ontogenetically young (up to 6 years). The growth strategies of *C. virginica* prevented varying degrees of time-averaging from being captured in the radiocarbon measurements at different points in the shell. This may not be the case for individuals that live longer, closer to 20 years. Thus, growth line analysis and age estimates are crucial when selecting samples.

Given that the spread in the uncalibrated ^{14}C dates is still larger than the ontogenetic age estimates for most samples, temporal variability in the local marine reservoirs and diet and metabolism effects may both be a source of the intrinsic intra-shell variability measured here (Lindauer et al., 2022; McConnaughey & Gillikin, 2008). Archaeologists often seek radiocarbon dates for the purpose of chronometric dates, in which case this variability poses a problem for producing secure dates. This approach operates on the assumption that the variability in the ^{14}C in the mollusk is characteristic of the variability in the marine reservoir throughout the mollusk's life (Jones et al., 2010), thus if the intra-shell variability is larger than the uncertainty on the ΔR value being used, then that ΔR value will not result in an accurate calibration of the shell.

The measured intra-shell radiocarbon variability may be more dramatic with more precise measurements. Jones et al. (2010) implements a more precise intra-shell measurements by milling within growth increments to obtain powder for radiocarbon analysis. This approach is often taken in studies that use intra-shell radiocarbon measurements to study changes in the marine reservoir (Culleton et al. 2006; Hadden & Cherkinsky 2017). However, even with less precise measurements and larger degrees of time-averaging, intra-shell variability can still be distinguishable in certain species and has consequences for archaeological applications of marine mollusk shell radiocarbon measurements even at a lower resolution.

Even though multiple ^{14}C measurements per shell were obtained, fabricated variability due to differing degrees of time-averaging also play a role in our data. For example, the chondrophore samples of *M. arenaria* and the hinge samples of *C. virginica* are effectively a lifetime average of the ^{14}C signal. In *S. gigantea* samples the carbonate from the umbo still contains signals from throughout the mollusk's entire life, but it contains more carbonate deposited earlier in life. This is visible in Figure 2.5 as the grouping of growth lines at the umbo becomes more dense with age.

The degree of time averaging in the ventral margin measurements also varies across the three species considered here. For *M. arenaria*, ventral margin measurements only represent the ^{14}C signal from the last few years of life. In our samples, very few growth lines were distinct in the ventral margin, and these samples only represent the very end of the mollusk's life and are the least susceptible to time-averaging. The ventral margin samples from *C. virginica* displayed growth line counts that were more challenging to interpret but appear to represent most of the mollusk's life. For *S. gigantea*, shell growth rapidly decreases as the mollusk's age increases, resulting in more dense groupings of growth lines at the ventral margin in older mollusks (Cannon & Burchell, 2009). Thus, the ventral margin samples from senile *S. gigantea* mollusks are more susceptible to time-averaging effects than the ventral margin of a species like *M. arenaria*.

In some situations, time-averaged radiocarbon dates are favourable for archaeological applications. Time-averaged measurements can be beneficial because they avoid anomalous radiocarbon measurements due to influxes of fresh water or dramatic upwelling events (Culleton et al., 2006, Hadden et al., 2023). However, taking samples that average over potentially centuries of growth will make the radiocarbon age appear much older. The chondrophore of *M. arenaria* and the hinge of *C. virginica* provide archaeologists the ability to obtain a time-averaged radiocarbon measurement without homogenizing the entire shell and allow for estimating ontogenetic age and thus the degree of time-averaging through growth line analysis. To ensure that decades of growth are not averaged over, growth line analysis should be completed to confirm the ontogenetic age of the mollusks and ensure short-lived samples are selected. This is particularly beneficial as the ventral margin is much more fragile and is not always preserved in archaeological contexts.

Further statistical analyses could be completed on these calibrated intra-shell dates include "wiggle-matching" which entails modeling dates sequentially based on their growth increments (Helama & Hood, 2011, Hadden et al., 2017). Both the precision and accuracy of the calibrated radiocarbon dates depend on the precision and accuracy of the ΔR values being used. Studies that address intra-shell radiocarbon variability such as this one are only a piece of the puzzle and should be complemented with rigorous analysis to improve ΔR corrections. For example, *C. virginica* is a viable species for radiocarbon analysis, but as Lelievre (2017) points out P.E.I. lacks ΔR corrections other than those from McNeely et al. (2006).

2.6 CONCLUSION

A comprehensive approach using radiocarbon analysis and growth line analysis was used to investigate intra-shell radiocarbon variability in *C. virginica* from Prince Edward Island, *S. gigantea* from British Columbia, and *M. arenaria* from Nova Scotia. We aimed to characterize the effects of fabricated variability due to sampling strategies by taking more than one measurement per shell as different growth strategies of mollusk species result in different degrees of time-averaging throughout a single shell. Factors contributing to fabricated variability can be limited by tailoring the sampling strategy to the mollusk's growth strategy and incorporating sclerochronological analysis - such as growth estimates - to ensure that the amount of time-averaging is accurately communicated.

Intrinsic variability — primarily referring to variability in the local marine reservoir over the mollusks lifetime which may be reflected in changes in ^{14}C throughout the shell — and fabricated variability are challenging to untangle and require high-resolution analysis of ^{14}C combined with other geochemical analyses (Jones et al., 2010, Hadden et al. 2017). For archaeological purposes using the readily available R_Combine function provides a quick method for analyzing the significance of the intra-shell radiocarbon variability.

In this work, *C. virginica* oysters from P.E.I. returned the tightest range of intra-shell radiocarbon measurements. All the oysters in this work were ontogenetically young, living only up to 6 years based on growth line analysis. Additionally, samples from both British Columbia and Nova Scotia failed the chi-squared test. Both British Columbia and Nova Scotia are subject to variable coastal circulation patterns which will impact the local marine reservoir effect and thus the degree of intra-shell radiocarbon variability. Archaeologists should proceed with caution when radiocarbon dating shell from these regions.

Our results reinforce the need to obtain multiple measurements per shell to properly assess the degree of radiocarbon variability in the shell and produce reliable calibrated dates (Hadden et al. 2017). These data could be supplemented further in future experiments by preserving half of the bivalve for a whole-shell average measurement — by homogenizing the entire shell and analyzing it for radiocarbon — and comparing to intra-shell measurements to further assess effects of sampling choices.

Additional work focusing on the contribution of metabolic ^{14}C for these species and species-specific ΔR corrections will further build confidence in these data and help researchers make informed choices on which samples are best to use for archaeological studies (Hadden et al. 2023). This study is only a preliminary look at a small subset of mollusk species present in archaeological contexts in Canada and we emphasize that a thorough consideration of growth strategies and biological factors of the mollusks present at any given site be considered to find the most suitable sample and measurement strategy.

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CHAPTER 3

REFINING THE RADIOCARBON CHRONOLOGY AT PORT JOLI HARBOUR, NOVA SCOTIA, CANADA WITH LOCAL ΔR AND MARINE SHELL DATES

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ABSTRACT

Port Joli Harbour, situated on the Southwest coast of the maritime province of Nova Scotia, Canada, has the highest density of shell midden deposits in Atlantic Canada. Previous excavations have yielded both marine and terrestrial materials suitable for radiocarbon analysis. The terrestrial chronology points to continuous occupation throughout the Middle-Late Woodland Periods (1685 calBP to 250 calBP), however marine shell samples have not been similarly studied. We report twelve radiocarbon measurements on the soft-shell clam *Mya arenaria* from the same depositional contexts as previously dated terrestrial samples. From these dates we construct a complementary marine chronology that shows good agreement with the terrestrial chronology. This builds confidence in the use of *M. arenaria* radiocarbon data in the region, especially from "ghost middens" — highly eroded middens that lack bone and charcoal, leaving only marine shells for radiocarbon analysis. We also report the first local marine reservoir correction (ΔR) calculations for the harbour using shell-charcoal and shell-bone pairs. The ΔR values show a variability of ± 150 years and are overall more positive than the pre-bomb values reported for the harbour (McNeely

et al. 2006). Confidence in marine shell radiocarbon measurements for *M. arenaria* in this region can be further improved by refining this value.

3.1 INTRODUCTION

Shell middens along the North Atlantic Coast of North America are an invaluable archive of past human-environment interactions in coastal settings. Port Joli Harbour (PJH) is a small inlet on the southeast coast of Nova Scotia, which contains 21 distinct shell midden deposits and represents the most dense concentration of shell middens in the region (Betts, 2019). PJH has been populated continuously for at least 1600 years, since the Middle Woodland Period, first by the pre-contact Mi'kmaq people and subsequently their descendants, the Mi'kmaq, who comprise the modern indigenous populations who live in the area (Betts et al., 2017; Betts, 2019). The E'se'get Archaeology Project, led by Dr. Matthew Betts, excavated and documented the stratigraphy, artifacts, and features found at several of the shell midden sites in PJH. Further building upon this foundational work, continued research has illustrated in detail the social, spiritual, and economic landscapes of PJH in the Middle to Late Woodland period, roughly 1000-2000 years ago, solidifying its place in Canadian archaeology as a significant coastal archaeology landscape (Betts et al., 2017; Hrynick & Betts, 2017).

The analysis of the marine mollusk shells from PJH midden sites can help archaeologists determine shellfish harvesting strategies of past populations, timing of site occupation, and provide a record of past sea surface temperatures in the harbour (Burchell et al., 2014). However, building confidence in marine shell dates is critical for anchoring palaeoclimate data in time and increasing the temporal precision of interpretations of human-environmental interactions with shellfish (Culleton et al., 2006; Deo et al., 2004; Hadden et al., 2023).

Despite the significance of the PJH shell midden sites for the study of ancestral Mi'kmaq populations and their relationship with the coastal environment which they inhabited, very little work has been done to build confidence in marine shell radiocarbon for this region. In particular, no archaeological local marine reservoir offsets (ΔR) have been calculated for this harbour specifically — a value which is necessary for calibrating marine dates. Other studies in Atlantic Canada that use radiocarbon measurements from marine shell samples, such as the study done by Lelièvre et al. (2017) in the Pugwash Basin in North West Nova Scotia, use a pre-bomb value for the calibration from McNeely et al. (2006). This paper presents the first investigation of ΔR values, including a consideration of temporal variability in these values, in PJH and explores the implications for building marine radiocarbon records for the region. By cross-referencing marine shell samples of the soft shell clam, *Mya arenaria*, from the collections at Memorial University with previously published terrestrial radiocarbon measurements (Betts, 2019), we construct a complementary marine chronology for the harbour to assess the agreement of the marine shell dates with the terrestrial dates. We also use the paired marine-terrestrial samples to calculate local marine reservoir offsets (ΔR) for the harbour and suggest steps for refining this value for wider use in Nova Scotia archaeology.

3.1.1 Geographic and Environmental Context of Port Joli Harbour

The coastal landscape of Port Joli and its surrounding regions have shaped and influenced the ancestral Mi'kmaq populations' food harvesting strategies. Considerations of the coastal nature of the environment of Port Joli has been central to most of the archaeological studies in the region, and the impact of this environment on the lifeways of the people inhabiting this region are crucial to understanding how the ancient Mi'kmaq in this area lived and thrived (Betts et al., 2017). Significant work using sediment cores and pollen records from Path Lake - located just under 1 km from the Harbour itself allowed for researchers to build a detailed record of highly localized environmental change in the region (Neil et al., 2014). The pollen records show evidence of the Little Ice Age - associated cooling in the harbour through the increased presence of boreal species between 900 and 260 cal BP, and periods of highest settlement intensity in the harbour also correspond to the highest annual precipitation levels, which likely led to lower salinity levels and higher nutrient loads in the harbour and a more favourable habitat for the soft shell clam, *Mya arenaria* (Neil et al., 2014).

PJH is located in southwest Nova Scotia and is a small inlet with sandy beaches surrounded by the Acadian-Boreal Coastal Forest (Neil et al., 2014). PJH is part of a larger region called the Scotian shelf, an oceanic region that encompasses much of the entire east coast of Nova Scotia and is influenced by several different ocean current and climate patterns such as the Gulf Stream, the Labrador Current, and the North Atlantic Oscillation, and Atlantic Multidecadal Oscillation (Deser et al., 2010; Lotze et al., 2022; MacLean et al., 2013).

3.1.2 Overview of Shell Midden Archaeology in Port Joli Harbour

Most of the midden sites in the harbour were first discovered by John Erskine in the mid 1900's (Erskine, 1959). The Harbour itself formed about 3000 years ago (see Neil et al., 2014), and the lack of archaeological sites dating prior to this date can be explained by their submergence as sea levels rose. Even amidst fluctuating sea levels, the coastal populations in Port Joli continued to utilize the landscape. For example, rising sea levels resulted in the creation of mudflats: an optimal environment for *Mya arenaria* to thrive and thus lead to the abundance of this resource for use by the coastal population. The location of these mudflats now corresponds with the location of shell midden sites around the harbour (Neil et al., 2014).

The largest intact midden in PJH, AlDf-24 was the focus of considerable attention between 2008 and 2010 (Betts, 2019). Excavation revealed that the site had the longest occupational history and deepest stratigraphic deposits and thus it yielded the most viable marine shell samples for radiocarbon analysis which were linked to terrestrial radiocarbon dates taken on bone and charcoal (Betts, 2019). Located on the South-West coast of the harbour, this midden features at least one dwelling area (Area C) and a shellfish processing area (Area A). Figure 3.1 shows the location of this site within the harbour.

Figure 3.1 Map of Port Joli Harbour, situating it within the wider context of the province of Nova Scotia, Canada. Key midden sites are labelled on the map, including the dwelling site AlDf-24.

AlDf-24C: Dwelling Area

AlDf-24C has several archaeological features that point to a well-established dwelling area. The deposit contains a variety of cultural materials including lithic tools and debitage, cultural charcoal, ceramics, burned mammal bone, and gray ashy soil interpreted as evidence of a hearth. Area C also has evidence of multiple stratigraphically superimposed dwelling floors, reinforcing that this was a site with repeated occupation. Level 2 in AlDf-24C was identified as an activity area, perhaps an ephemeral house floor, which was used for producing stone tools due to the abundance of lithic tools and debitage found in this level.

The terrestrial dates in this midden also reinforce continuous use as a dwelling space as the three terrestrial dates from this midden span a much larger time frame compared to other middens in the harbour. The basal cultural date (point 1758 in Figure 3.4 A) dates from the beginning of the Late Maritime Woodland period at 660-880 calAD, and the uppermost layer (point 1757 in Figure 3.2 A) dates to the Proto-Historic period at 1440-1640 calAD.

AlDf-24A: Shellfish Processing Area

In comparison with AlDf-24C, radiocarbon dates from AlDf-24A spanning the depth of the midden (samples points 1748, 1745, 1749, and 1769 in Figure 3.2 B) reported a much narrower range: from 430 calAD to 660 calAD. This narrower range of dates suggests the formation of this midden was quite rapid, which would be consistent with a site that was used intensely as a processing site for shellfish. Given the fast accumulation, this site makes for a valuable point of comparison with marine dates from the same midden since the terrestrial dates span so little time.

AlDf-24A also contained a much larger amount of shell material compared to cultural material, likely because this midden was used for drying shellfish and cooking other meat for consumption and was mainly for food processing and preparation rather than a shared dwelling space. The significance of the lower layers of the midden stratigraphy are not entirely clear. Level 3m of AlDf-24A represents a lower layer of the midden, one layer above the lowest cultural layer (3n). Upper layers contain more cultural materials like ceramic sherds and a variety of faunal remains. Level 3m contained primarily shell fragments and some sparse ceramic pottery sherds and fish bones. Level 3n is the lowest cultural layer of AlDf-24A, identified as such by a few lithic artifacts. The lithic artifacts present in layer 3n are likely a result of disturbance and interlayer mixing from above layers, which is evidenced by the presence of crushed shells from trampling.

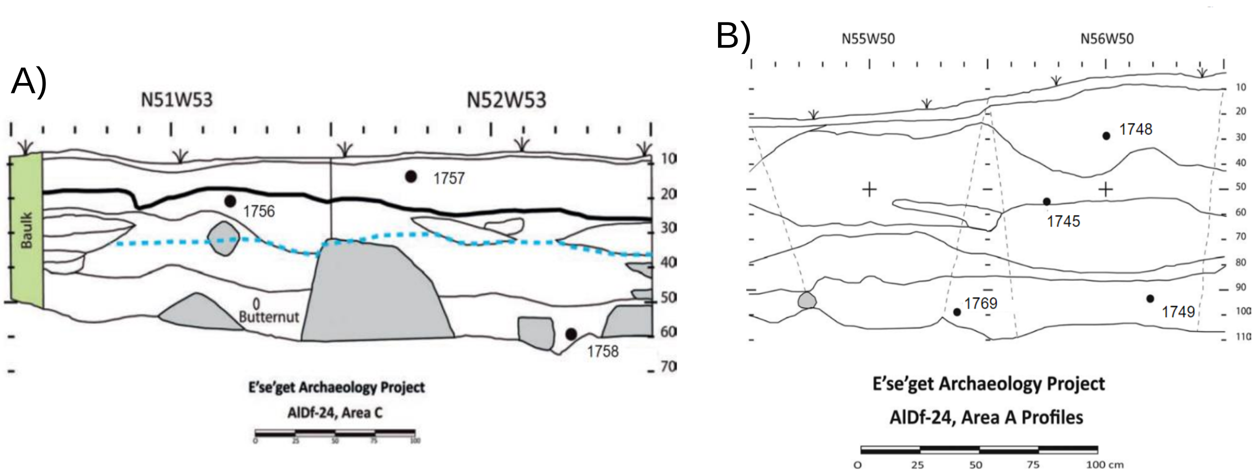

Figure 3.2 Images reproduced from Betts (2019) showing the stratigraphic profile of AlDf-24 Area C (subfigure A) and Area A (subfigure B). The numbers indicate the sample IDs in Betts (2019).

Overview of Port Joli Harbour Radiocarbon Chronology

The differentiation of the Middle, Early Late, and Later Late Maritime Woodland periods correspond to characteristic ceramic trends and was first proposed by Black (Black, 1989) who correlated these trends with the work of Petersen and Sanger (1991). The date ranges for these periods are as follows: Middle Maritime Woodland from 250

B.C. to 650 A.D., Early Late Maritime Woodland from 650 A.D. to 1000 A.D., and Later Late Maritime Woodland from 1000 A.D. to 1400 A.D.

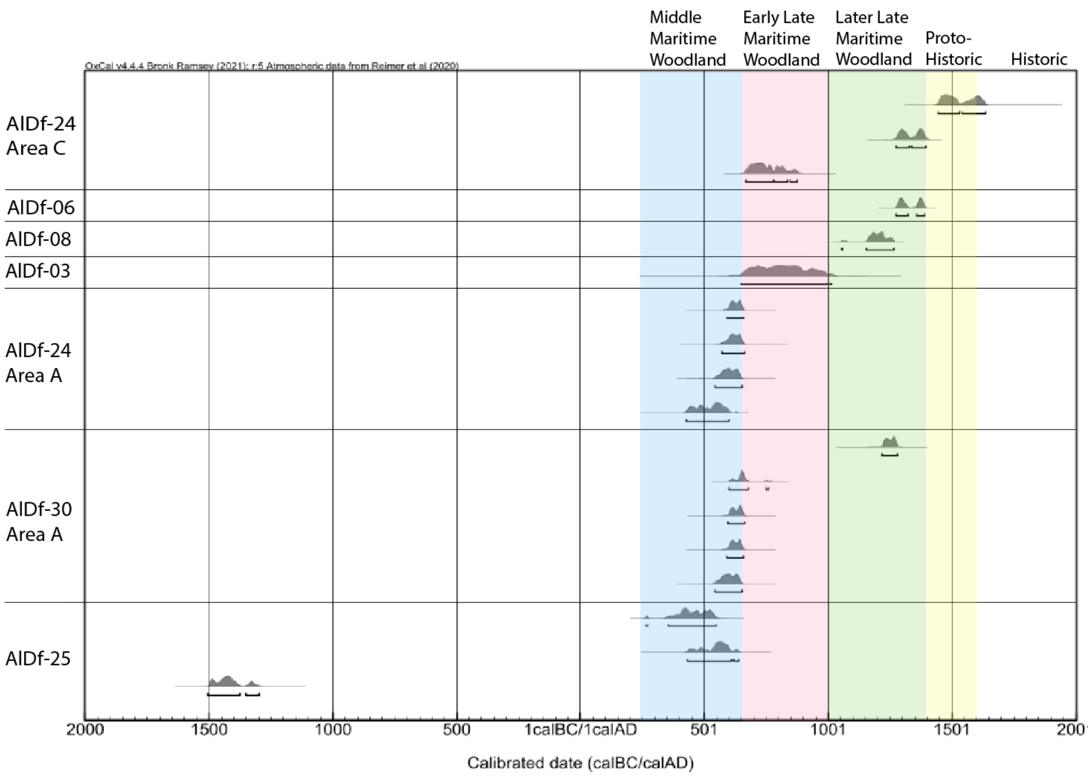

Figure 3.3 Terrestrial chronology for Port Joli Harbour. Dates were initially published in Betts (2019), Figure 4.1 from that publication has been reproduced here to with calibrations updated to the IntCal20 curve.

From the excavations done during the E'se'get archaeology project, a sequence of 18 radiocarbon dates were obtained on terrestrial mammal bone and charcoal samples from various midden sites around the harbour. These data were first published by Betts (2019), with dates calibrated to the IntCal09 curve. The chronology is reproduced in Figure 3.3, with all dates recalibrated to the IntCal20 curve. The updated terrestrial chronology is still consistent with continuous occupation throughout the Middle to Late Woodland periods and into the Proto-Historic. The much older date returned from AlDf-

25 is from a charcoal sample, but analysis of the pottery places the context of the charcoal sample in the Middle Maritime Woodland Period, suggesting that this date may not be cultural (Betts, 2019).

3.1.3 *Mya arenaria*: The Soft-Shell Clam

The most abundant species of mollusk found in the middens at Port Joli is the soft-shell clam *Mya arenaria*. The relevant biological and life history information, as well as the use of this species in previous archaeological studies is outlined below.

Life History Traits and Shell Structure of Mya arenaria

M. arenaria, has a large ecological range, extending along both the Pacific and Atlantic coasts of North America and the Atlantic coast of Europe as well as low- salinity areas in Europe (Heibert, 2015). *M. arenaria* entails a rapid growth period during the summer months when nutrients are abundant and a slow, nearly stagnant, period in the winter months when nutrients are more scarce, thus forming annual growth lines (DFO, 1985) (see Figure 3.4). The average life-span of this species is reported to be 10-12 years, but individuals have been recorded to live up to 28 years, with decreasing growth rate as age increases (DFO, 1985; Heibert, 2015; Strasser, 1999).

The shell of *M. arenaria* is entirely composed of calcium carbonate ($CaCO_3$) in the form of aragonite. Two distinct microstructures of aragonite have been identified in *M. arenaria*: prismatic and crossed-lamellar, with some regions of the shell displaying alternating layers of crossed-lamellar and prismatic (De Noia et al., 2020). As with many other bivalve mollusks, the chemical composition of the shell of *M. arenaria* reflects that of the marine environment in which it was biomineralized. The chondrophore of *M. arenaria* contains a complete record of growth increments and thus a complete record of the marine environment throughout the mollusk's life whereas the ventral margin contains only the most recent growth records and is more subject to remodeling.

Figure 3.4 Cross section of *Mya arenaria* chondrophore with annual growth lines visible. Dark bands correspond to cold periods of slowed growth and light bands correspond to warm periods of more rapid growth. Credit: Ian Predham.

Archaeological Significance of Mya arenaria

Several other archaeological sites throughout the Atlantic Coast of North America also report substantial amounts of *M. arenaria* shells in archaeological midden deposits (Ambrose et al., 2016; Blackwood, 2019; Lelièvre, 2017). More recent work has begun to highlight the importance of this mollusk species in archaeological studies in the North Atlantic. An initial study on the application of growth increment analysis to determine season of harvest has been completed at Port Joli (Burchell et al., 2014; Betts et al. 2017) and similar studies have been completed in the Gulf of Maine (Ambrose et al., 2016;

Blackwood, 2019; Lightfoot et al., 1993). More detailed biological studies on growth variability will find further use in archaeological contexts in order to improve age estimates (Koo et al., 2017). However, the use of this species in marine ^{14}C studies in the region is still limited, but in recent years *M. arenaria* has been suggested to be a good candidate for marine reservoir studies (Lelièvre, 2017; Lotze et al., 2022).

3.2 MATERIALS AND METHODS

To investigate the agreement between marine and terrestrial radiocarbon measurements and to calculate ΔR values for PJH, previously excavated M. arenaria shells were sorted to identify samples that matched the depositional contexts of the previously dated terrestrial samples from Betts (2019). Shell samples that were the most intact were chosen and dating shell fragments was avoided so that we could preserve as much material as possible for further analysis.

3.2.1 Pairing Terrestrial and Marine Samples

The terrestrial samples reported by Betts (2019) were derived from a specific stratigraphic layer and can be associated with a minimum 5 cm vertical increment within that layer. Shell samples that were selected during the 2021 inventory at Memorial University were securely associated with stratigraphic layers that align with those of the terrestrial samples. These pairs were identified based on previously excavated samples that were readily available for analysis. Nine pairs were identified during the inventory and the five pairs with the most intact shells were selected for analysis. The five pairs are discussed below.

Pair 1

Pair 1 originates from the lowest secured cultural layer in AlDf-24A - level 3m. This layer contains two pottery sherds, as well as shell and bone remains which were the result of human activity, warranting the description as a cultural layer. Shell in this layer was fragmented, but we recovered a shell with an intact chondrophore and slightly chipped ventral margin. The terrestrial sample from this level is a charcoal sample, labeled as 1749 in Figure 3.2 B).

Pair 3

Pair 3 originates from the basal level in AlDf-24A - level 3n. This level was identified as separate from level 3m because it is primarily composed of crushed shell intermixed with soil, likely because the cultural material in this level has been packed down from the above level 3 m. From this layer we recovered a shell fragment with an intact chondrophore and a slightly chipped ventral margin. The terrestrial sample from this level is a charcoal sample, labeled as 1769 in Figure 3.2 B).

Pair 6

Pair 6 originates from Level 2 in AlDf-24C. This level was identified as an activity area because of the abundance of debitage from stone tool making activities, as well as an abundance of bone and charcoal. Shell samples were sparse from Level 2, but we recovered a sample mostly intact, with some chips on the ventral margin and chondrophore. The terrestrial sample from this level is caribou tooth, labeled as sample 1757 in Figure 3.2 A).

Pair 8

Feature 4 in AlDf-24C identifies deep floor surfaces of a dwelling that was repeatedly used over time. It was identified as a house floor due to its very compact soil, less (and highly fragmented) shell material than in other deposits, and more artifacts and lithic remains. The caribou bone was excavated from a hearth feature in Feature 4 and was identified to be contemporaneous with the dwelling floor (levels 3 and 3b) of Feature 4. The terrestrial sample from this level is caribou bone, labeled as sample 1756 in Figure 3.2 A).

Pair 9

Level 3i contains shell material and some artifacts and is situated directly below artifact rich layers with an abundance of whole shells. The compact matrix of sandy-loam in this layer, coupled with the observed gradual transition from upper subdivisions of layer 3 suggest that the shell and disperse artifacts present are representative of the layers above. Thus, Betts groups levels 3f – 3i as being contemporary. From the collections at Memorial University, we were able to find 3 shells that could be grouped with a terrestrial caribou bone date from AlDf-24C, level 3i. The shell recorded as Pair 7 has also been grouped with terrestrial Pair 9. The terrestrial sample from this level is caribou bone, labeled as sample 1758 in Figure 3.2 A.

3.2.2 FT-IR

Fourier Transform Infrared Spectroscopy (FT-IR) probes the vibrational modes within a sample by analyzing how the sample interacts with infrared radiation. Depending on the composition and structural properties of the material, different frequencies of infrared radiation will be absorbed by the sample and will appear as peaks in the FT-IR spectrum. Despite having the same chemical formula, calcite and aragonite have different absorption peaks in the mid infrared range due to their different crystal structure. This makes FT-IR a good candidate for detecting the dissolution and reprecipitation of aragonite as calcite. Re-precipitated calcite from aragonite marine samples can incorporate new carbon from the marine environment, causing erroneous ^{14}C measurements that do not accurately reflect the age of the mollusk shell.

Shell material was taken from the shells by scratching the surface of the shell with a metal scoop. Powder was collected from the inner and outer portions of the ventral margin for all samples and from the chondrophore for samples AlDf-24A-1, AlDf-24C-9b, and AlDf-24C-8c. FT-IR data was collected with the Bruker Alpha II spectrometer. Chondrophore samples were analyzed using the attenuated total reflectance attachment (see more details in Appendix A.1) and all ventral margin samples were analyzed using the transmission attachment (see more details in Appendix A.2).

3.2.3 Shell Preparation and Sampling for Radiocarbon Dating

The ventral margin of the *M. arenaria* samples were cut using a small electric saw to obtain a sample about 1 cm by 1 cm, capturing only the last few years of growth of the mollusk. A Dremel was used to cut the chondrophore from the shell with the aim of preserving the entire chondrophore for radiocarbon measurements.

The selected shell samples were prepared and analyzed for ^{14}C by laboratory technicians at the Lalonde AMS laboratory from the University of Ottawa in December of 2021 following the preparation procedures outlined in Crann et al. (2017). All samples were pre-etched in HCl to remove the outermost 20% of the shell $CaCO_3$, ensuring that any surface contaminants were removed. Following the acid etch, CO_2 gas, the carbon of which is derived from the carbon from the shell carbonate, is produced by reacting the carbonate with phosphoric acid (H_3PO_4). The CO_2 collected from this process is then reacted with hydrogen in the presence of iron to produce graphite – elemental carbon – which is the final product in the preparation process (Crann et al., 2017). Graphite is then analyzed by means of accelerator mass spectrometry to determine its isotopic composition. Samples were corrected for fractionation by normalizing $^{13}C/^{12}C$ values to -25 with respect to Pee Dee Belemnite. Sample activities were normalized to the Oxalic Acid II standard (Crann et al., 2017; Stuiver & Polach, 1977).

3.2.4 ΔR Calculation

The generalized equation for calculating a ΔR value is given as:

$$\Delta R = P - Q$$

Where P is the measured conventional age of a marine radiocarbon sample, and Q is the expected conventional age of the sample. The calculation of the local marine reservoir offset was done using an open access online ΔR calculator developed by Ron

Reimer and Paula Reimer (R. W. Reimer & Reimer, 2017). This tool is designed to account for the errors in both the terrestrial and marine radiocarbon dates by using a convolution integral to determine a confidence range for the offset between the two measured ages. The tool gives the option to input data as either a calendar age (i.e. a collection date), or a measured radiocarbon age (uncalibrated years BP).

The method implemented in this online application uses the full probability distributions of the calibrated ranges to produce the probability ranges of the ΔR measurement, which returns a more realistic uncertainty on the ΔR measurement than the intercept method (R. W. Reimer & Reimer, 2017). The steps outlined by Reimer & Reimer (2017) method used for paired marine and terrestrial samples involves the following steps:

1. Calibrate the terrestrial ^{14}C measurement to the appropriate terrestrial calibration curve (either Northern or Southern Hemisphere calibration curve), in this case the Northern Hemisphere curve.

2. Reverse calibrate this measurement to the marine calibration curve using discrete points from the probability density function of the calibrated date produced in step 1, the resulting value is denoted as *Marine*20*Ct*.

3. ΔR is then calculated by subtracting the corresponding Marine20 age at that time (*Marine*20*Ct*) from the measured radiocarbon age of the corresponding marine sample (14*Cm*), as shown in the following equation:

$$\Delta Rt = 14Cm - Marine20Ct.$$

3.3 RESULTS

FT-IR analysis was used to ensure that samples of *M. arenaria* were pristine aragonite and did not have any evidence of calcite reprecipitation. The AMS radiocarbon dates obtained from the *M. arenaria* shells are used to construct a complementary marine chronology by calibrating the dates using a pre-bomb ΔR for the region and the marine-terrestrial pairs are further used to calculate archaeological ΔR values for the harbour.

3.3.1 FT-IR

All samples from *M. arenaria* were found to be consistent with aragonite, and no evidence of contaminating calcite. The spectrum in Figure 3.5 shows a transmission FT-IR spectrum from shell AlDf-24A-1 with the characteristic peaks labeled. See supplementary information for additional infrared spectra.

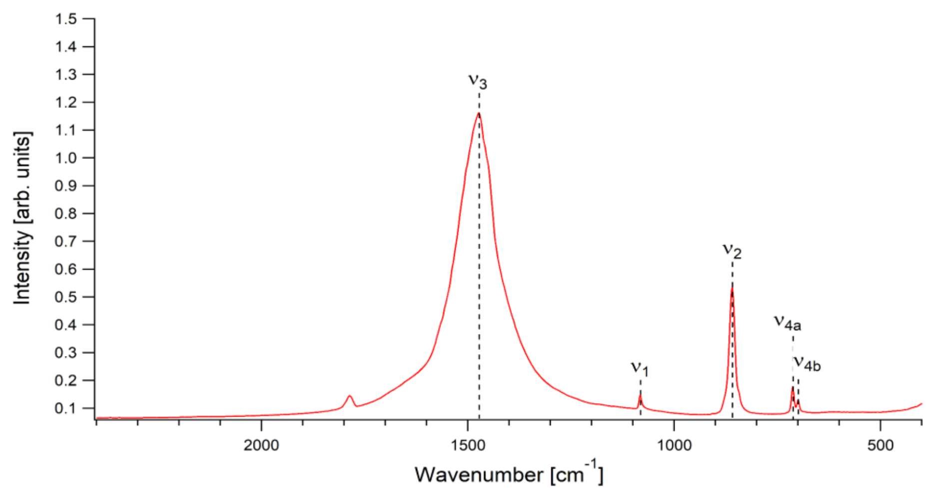

Figure 3.5 Infrared spectrum of pristine aragonite from sample AlDf24A-1. The peaks labeled on the plot correspond to specific vibrational modes of the CO_3^{2-} moiety as follows v1: symmetric stretching, v2: out-of-plane bending, v3: asymmetric stretching, and v4: in-plane bending. The peak at 1785 cm^-1 is a combination mode of symmetric stretching and in-plane bending (v1+v4) and is characteristic of aragonite.

3.3.2 Radiocarbon and ΔR

The radiocarbon measurements from the marine shell samples and their corresponding terrestrial pairs were used to calculate ΔR values for the harbour. Table 3.1 reports the raw, uncalibrated fraction of modern carbon (F14C) from each of the samples with its corresponding reported error. The corresponding calibrated corresponding terrestrial values from Betts et al. (2019) have been updated with the IntCal20 calibration curve. Twelve ΔR values are reported for the harbour and three shell samples (AlDf24A-1, AlDf24C-9b, and AlDf24C-8c) were used to investigate the effect of sampling location on the calculated ΔR value by taking one radiocarbon measurement at the ventral margin and one at the chondrophore. The sample location is denoted in the table by either a "C" for chondrophore, or "VM" for ventral margin.

The ΔR values reported in Table 3.1 are plotted against the corresponding terrestrial age in Figure 3.6. The twelve ΔR values calculated for PJH range from -40 years to 314 years. Calculations using samples from the chondrophore consistently result in more positive ΔR values, however variability between shells is more significant than variability resulting from different sampling locations within the same shell. In Figure 6, pairs which are from AlDf-24A and those from AlDf-24C are distinguished. As discussed previously, AlDf-24A is thought to have deposited very rapidly due to the terrestrial dates spanning the midden all clustering together, whereas AlDf-24C was repeatedly occupied for a period up to 1000 years in length. Despite the suspected short accumulation period of AlDf-24A, the ΔR values are no less variable than those reported from AlDf-24C.

Following the procedure outlined by Martindale et al. (2018), a weighted mean ΔR value was calculated as follows:

1. Calculate ΔR values from the convolution integral method implemented in the deltaR online tool.

2) Calculate the error on the weighted mean of the ΔRs from all possible pairs

3) Calculate the standard deviation of the ΔR values

4) Calculate the standard error by computing the square root of the sum of the squares from step 2) and 3).

Table 3.1 Radiocarbon data from 11 *M. arenaria* shells from Port Joli. The conventional radiocarbon ages are reported with their uncertainties. Terrestrial values were calibrated using the IntCal20 curve and ΔR values were calculated using the online tool. Both 1σ and 2σ confidence ranges the ΔR values are reported.
Note that **δ^{13}C** values for shell samples from Lalonde are not reported as they contain machine fractionation (Crann et al. 2017).

Pair	**Lab ID**	**Other ID**	**Material**	**Sample location**	**δ^{13}C**	**14C yr BP**	**±**	**ΔR (years, 2σ)**	**ΔR (years, 1σ)**
1	1749	273513	Charcoal	-	-23.9	1470	40	-	-
	UOC-18192	AlDf24A-1-c	Shell	C	-	1965	11	**-5 ± 64**	0 ± **34**
	UOC-18193	AlDf24A-1-v	Shell	VM	-	1947	11	-23 ± **64**	-18 ± **34**
3	1769	297214	Charcoal	-	-23.8	1420	30	-	-
	UOC-18194	AlDf24A-3-v	Shell	VM	-	2162	11	223 ± **46**	224 ±**24**
6	1757	288732	Caribou tooth	-	-22.6	380	40	-	-
	UOC-18195	AlDf24C-6-v	Shell	VM	--	984	11	10 ± **104**	9 ± **85**
8	1756	286106	Caribou bone	-	-21.7	660	40	-	-
	UOC-18200	AlDf24C-8b-v	Shell	VM	-	1513	11	290 ±**75**	288±**61**
	UOC-18201	AlDf24C-8c-c	Shell	C	-	1358	11	135 ± **75**	133±**61**
	UOC-18202	AlDf24C-8c-v	Shell	VM	-	1352	11	129 ± **75**	127 ± **61**
	UOC-18203	AlDf24C-8d-v	Shell	VM	-	1320	11	97±**75**	95±**61**
9	1758	288733	Caribou bone	-	-20.8	1260	40	-	-
	UOC-18197	AlDf24C-9a-v	Shell	VM	-	1914	11	123±**102**	111±**72**
	UOC-18198	AlDf24C-9b-c	Shell	C	-	1722	11	-69±**102**	-81±**72**
	UOC-18199	AlDf24C-9b-v	Shell	VM	-	1684	11	-107±**102**	-119±**72**
	UOC-18196	AlDf24C-7-v	Shell	VM	-	1703	11	-88±**102**	-100±**72**
Weighted Mean Following Martindale et al. (2018)									94±**30**

Other ID	Borden	Area	Feature	Unit	Level	North,West	Depth (cm)
273513	AlDf-24	A	N/A	N56W50	3m	67,53.5	88
AlDf24A-1-c	AlDf-24	A	N/A	TP1 (N56W50)	3m	-	-
AlDf24A-1-v							
297214	AlDf-24	A	N/A	N55W50	3N/0, Dates basal level	83.5,18.5	89
AlDf24A-3-v	AlDf-24	A	N/A	TP1 (N56W50)	3n	53,51	99
288732	AlDf-24	C	Level 2 Activity Area	N52W53	2	SE Quad, SE Quad	Level Bag 8-14 cm
AlDf24C-6-v	AlDf-24	C	4	N53W53	2	NW Quad	-
286106	AlDf-24	C	4a	N51W52	I -Dates levels 3 and 3b in Feature 4	68,79	16.5
AlDf24C-8b-v	AlDf-24	C	4	N51W52	3	SE Quad	-
AlDf24C-8c-c	AlDf-24	C	4	N52W54	3	NW Quad	-
AlDf24C-8c-v							
AlDf24C-8d-v	AlDf-24	C	4	N52W51	3b	NW Quad	-
288733	AlDf-24	C	N/A	N52W53	3i - Dates level 3f-3k in Area C	NE Quad	50-73
AlDf24C-9a-v	AlDf-24	C	N/A	N52W53	3i	NE Quad	-
AlDf24C-9b-c	AlDf-24	C	N/A	N52W51	3h	NW Quad	-
AlDf24C-9b-v							
AlDf24C-7-v	AlDf-24	C	4	N52W51	3i	NE Quad	-
[redacted]							

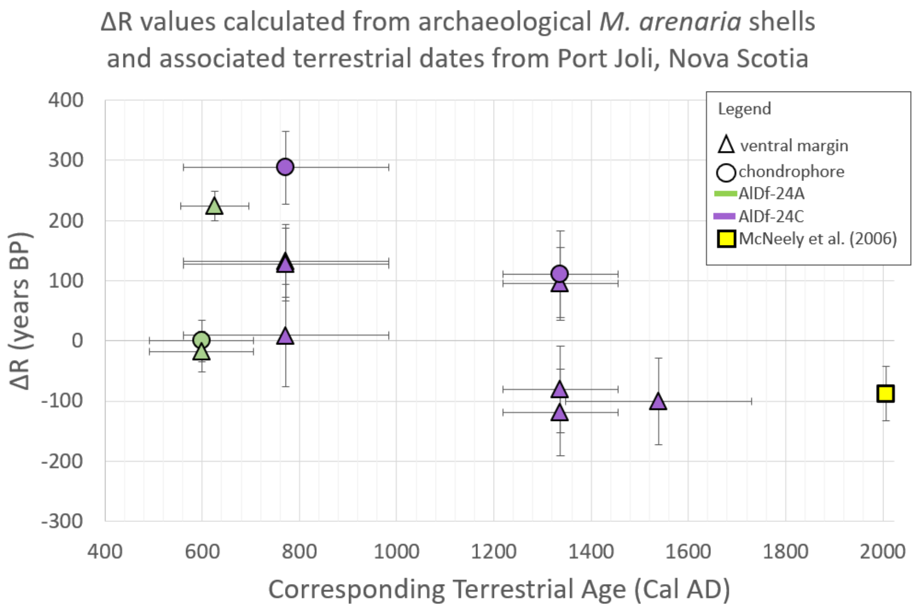

Figure 3.6 ΔR calculations (2σ) using archaeological *M. arenaria* shells plotted against their corresponding terrestrial dates. The sampling location within the shell is denoted by the shape of the markers and the color of the markers denotes whether the marine-terrestrial pairs were located within AlDf-24A, the rapidly deposited processing midden, or AlDf-24C, which has evidence of roughly 1000 years of occupation.

These steps were completed using the spreadsheet from the supplementary materials provided by Martindale et al. (2018) using the 1σ ΔR values as recommended by the authors. Table 3.3 shows that this analysis results in an estimated average ΔR for Port Joli of 94 ± 30 years for all the ΔR values reported in Table 3.3.

Pre-bomb ΔR values are often used to calibrate marine radiocarbon measurements when archaeological values are not available. To investigate how a fully marine chronology may shift or expand the existing terrestrial chronology, our marine radiocarbon data from *M. arenaria* are first calibrated to the Marine20 calibration curve (Heaton et al., 2020) using a value ΔR of -88±45 yrs, which was taken as the average of the 5 closest ΔR values near PJH as reported on the chrono ΔR database. These data were first reported in the 2006 Geological Society of Canada (GEOSCAN) survey and were calculated from live-collected specimens between 1908 and 1950 from between 26-76 km from the harbour (McNeely et al., 2006; P. J. Reimer, n.d.). These dates are also calibrated to our archaeological ΔR value of 94 ± 30 yrs to illustrate the effects of an updated ΔR on the marine calibrations. The results from both calibrations are reported in Table 3.4 and the calibrated age distributions are shown in stacked plots in Figure 3.7 and Figure 3.8.

Table 3.4 Radiocarbon dates from archaeological *M. arenaria* shells calibrated to the Marine20 calibration curve using the average of the 5 nearest pre-bomb ΔR values from McNeely et al. (2006) and our calculated archaeological ΔR value.

Pair	Sample Code	Sample location	14C yr BP	±	Marine calBP (2σ, ΔR= -88 ± 45)	Marine calBP (2σ, ΔR=94 ± 30)	Corresponding terrestrial date (2σ, calBP)
1	AlDf24A-1-c	C	1965	11	1452 ± 158	1258 ± 133	1352 ± 54
	AlDf24A-1-v	VM	1947	11	1436 ± 153	1241 ± 133	
3	AlDf24A-3-v	VM	2162	11	1684 ± 173	1465 ± 145	1325 ± 35
6	AlDf24C-6-v	VM	984	11	503 ± 141	354 ± 137	412 ± 96
8	AlDf24C-8b-v	VM	1513	11	996 ± 167	799 ± 129	614 ± 60
	AlDf24C-8c-c	C	1358	11	822 ± 149	657 ± 124	
	AlDf24C-8c-v	VM	1352	11	816 ± 146	652 ± 122	
	AlDf24C-8d-v	VM	1320	11	794 ± 141	623 ± 115	
9	AlDf24C-9a-v	VM	1914	11	1404 ± 149	1205 ± 133	1179 ± 106
	AlDf24C-9b-c	C	1722	11	1197 ± 149	1029 ± 143	
	AlDf24C-9b-v	VM	1684	11	1152 ± 153	979 ± 152	
	AlDf24C-7-v	VM	1703	11	1181 ± 152	1010 ± 150	

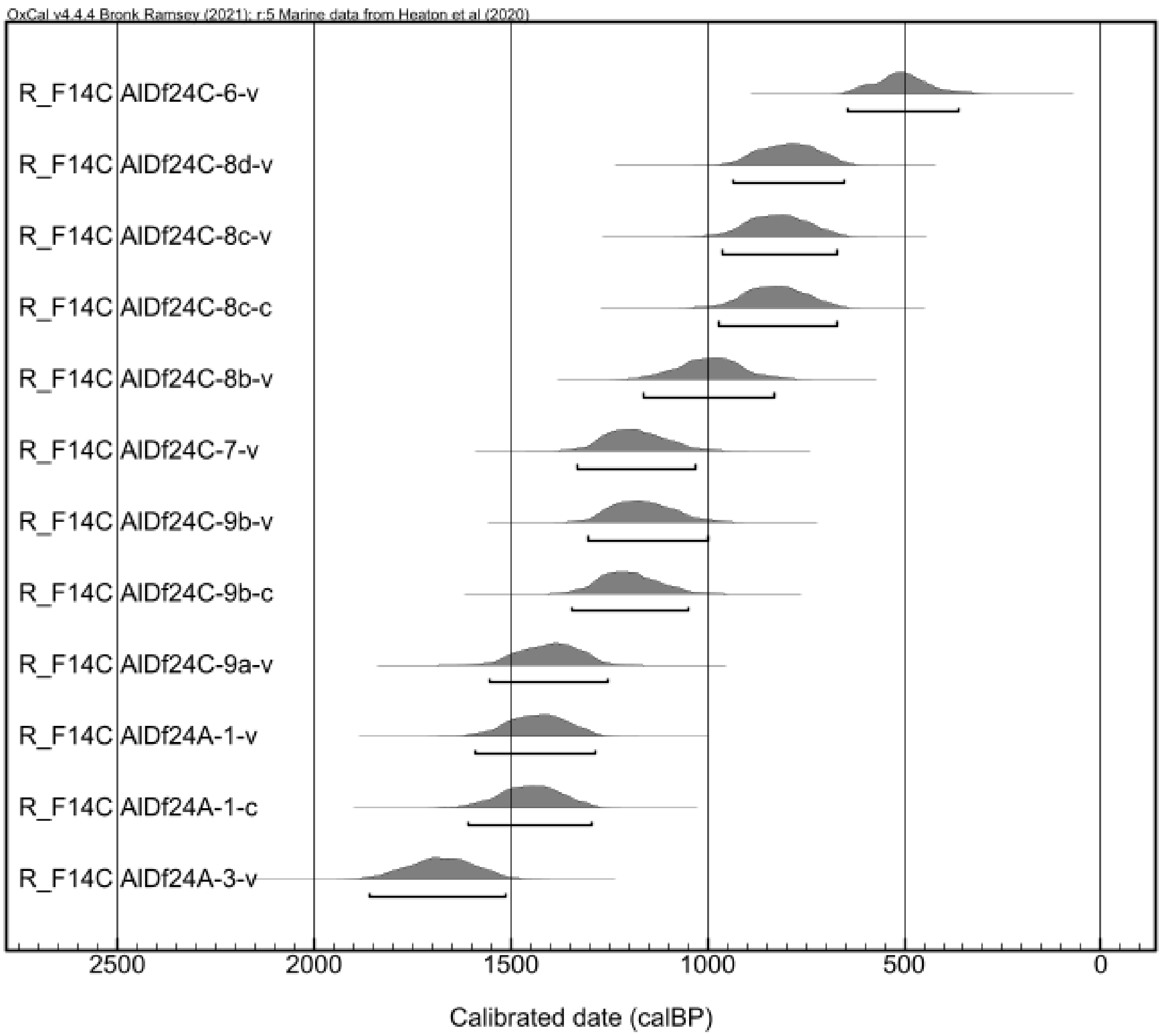

Figure 3.7 Complementary marine chronology for Port Joli Harbour. All dates are from *M. arenaria* shells and are calibrated to the Marine20 curve using a pre-bomb ΔR value of -88±45 (McNeely et al. 2006).

Both variations of the complementary chronology (with a pre-bomb ΔR and with our archaeological ΔR) reinforces continuous occupation throughout the entire Middle-Late Woodland Period in Port Joli. Further, the dates from AlDf-24A confirm a relatively rapid formation period, while the dates from AlDf-24C confirm an extended period of

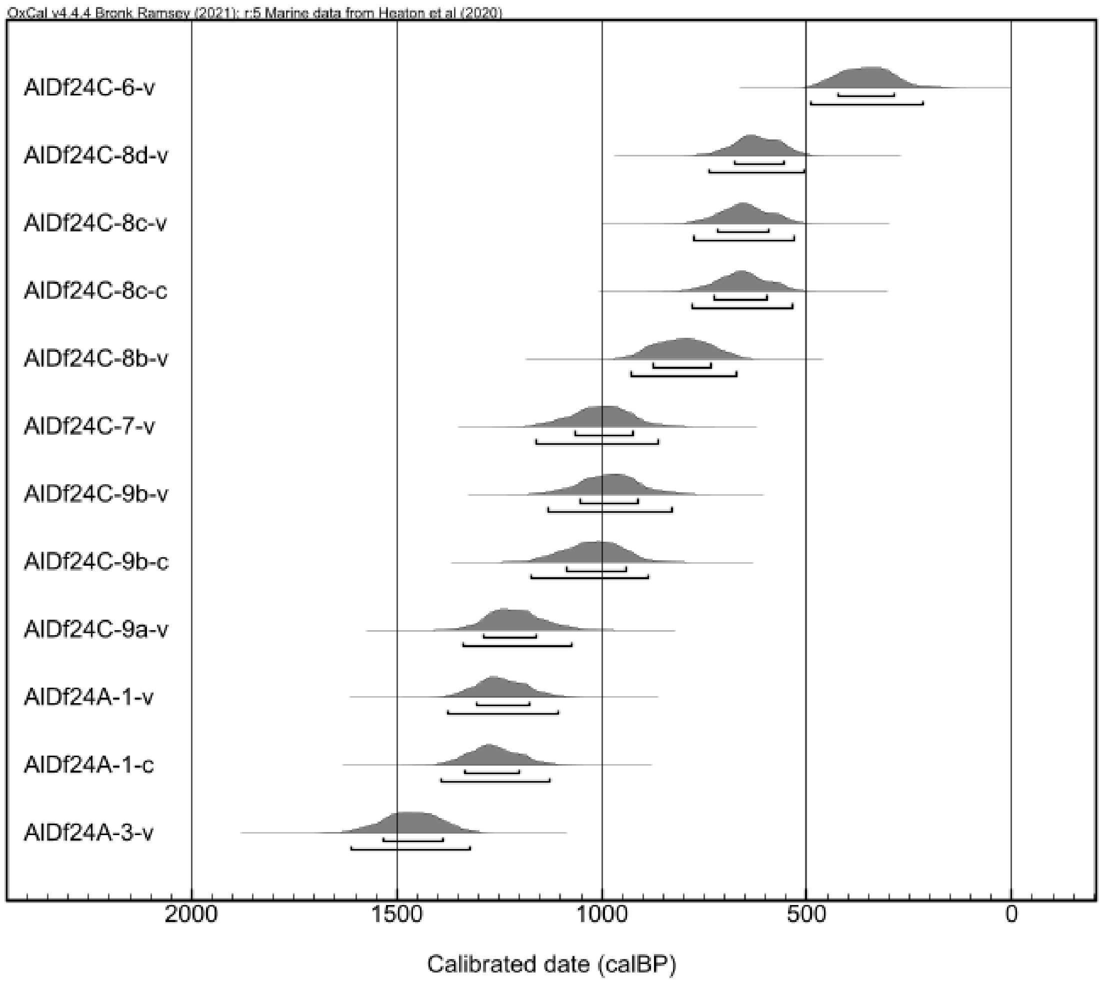

Figure 3.8 Radiocarbon dates from *M. arenaria* shells calibrated to 2σ using our calculated ΔR of 94 ± 30 yrs.

repeated or continuous occupation. Two of the marine samples calibrated with a pre-bomb ΔR do not overlap with the corresponding terrestrial sample within the 2-sigma probability range: AlDf24A-3-v and AlDf-4C-8b-v. However, all the calibrated values using the archaeological value of 94±30 yrs overlap within their uncertainties. While there are some discrepancies between the marine and terrestrial radiocarbon values for some of the pairs, the results do show reasonable correlation which can likely be improved as the ΔR values for the harbour are refined. Lastly, the *M. arenaria* dates were re-calibrated using our calculated ΔR value to assess how an updated ΔR value will impact interpretations from the marine chronology.

3.4. DISCUSSION

We first discuss the agreement of the *M. arenaria* dates with the previously reported terrestrial dates. These data show good agreement with the terrestrial chronology when calibrated with pre-bomb ΔR values that are currently available and with our archaeological ΔR value calculated from the marine-terrestrial pairs. The calibrated ranges with archaeological ΔR values have much larger uncertainties, but still overlap with all of the contemporaneous terrestrial values. The variation in the archaeological ΔR values calculated from the paired marine-terrestrial samples suggests that the accuracy of marine radiocarbon dates from PJH can be further improved by refining this value. This may also open avenues to study changes in the local marine environment in PJH by analyzing ΔR more thoroughly to investigate changes in the marine reservoir over time and connect this variability to broader climate changes.

3.4.1 Complementary Marine Chronology

The *M. arenaria* dates were first analyzed by calibrating them using an existing pre-bomb ΔR value. The McNeely et al. (2006) Geological Society of Canada (GEOSCAN) survey from 2006 contains a comprehensive database of marine samples of known age which were analyzed for radiocarbon to produce a bank of ΔR values spanning all coasts of Canada. Within the region of Eastern Nova Scotia, several pre-bomb ΔR values are reported in the GEOSCAN survey. The five samples closest to Port Joli as determined by the online ΔR tool (R. W. Reimer & Reimer, 2017) were selected for this statistical analysis and are reported in Table 3.5. Only these five data points were chosen because their geographic proximity to Port Joli was the closest and are all located along the south-eastern coast of Nova Scotia, in the same oceanographic environment along the Scotian shelf. For the raw data, see Appendix A.3.

The oldest cultural date reported by Betts (2019) was from site AlDf-30, and dates to 1544 ± 140 calBP and the oldest date from AlDf-24A from Betts (2019) dates to 1352 ± 54 calBP (both shown in Figure 3.9). The oldest date reported in this work is from AlDf-24A and dates to 1684 ± 173 calBP using the pre-bomb ΔR value reported above (shown in Figure 3.10). Comparing to the pre-bomb ΔR calibration, there could be as much as a 200 year shift in the starting date for the occupation of the harbour and up to a 300 year shift for the formation of the AlDf-24A midden. However, all of the calibrated ranges overlap and the calibrations with the updated ΔR value of 94 ± 30 yrs is in

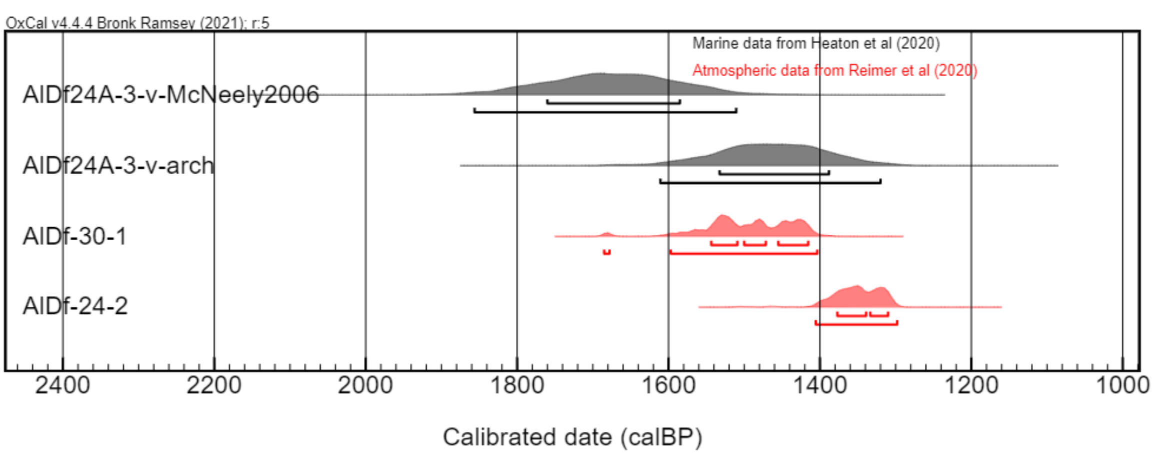

Figure 3.9 Oldest *M. arenaria* dates reported in this work calibrated with a pre-bomb ΔR value (-88 +/- 45 yrs)(McNeely et al. 2006) and calibrated with our archaeological ΔR value (94+/-30 yrs), compared to AlDf-30-1 (the oldest date in Port Joli Harbour reported by Betts (2019)) and AlDf-24-2 (the oldest date from site AlDf-24A reported by Betts (2019)).

agreement with the terrestrial dates (see Figure 3.9). This possible shift needs to be corroborated with more samples — preferably both marine and terrestrial — and ideally a more refined ΔR value that does include charcoal samples so as to avoid the old wood effect.

Although there are some discrepancies between the terrestrial and marine dates, the similarities between the two chronologies are apparent, with the marine dates from AlDf-24C confirming long-term, repeated occupation and the dates from AlDf-24A confirming rapid deposition. Calibrating the values using a pre-bomb ΔR value, as we have done here, does not introduce significant differences in the marine chronology compared to the terrestrial chronology. This is an encouraging result for future work using radiocarbon analysis at midden sites that have been subject to coastal erosion which has caused damage to other cultural material like charcoal and bone or washing them away completely, leaving shell as the only viable material for radiocarbon analysis. These types of middens, referred to as "ghost middens", have recently been identified in neighbouring harbours to PJH in Southwestern Nova Scotia (Betts, 2022). Additionally, many of the middens on the west coast of PJH are more susceptible to coastal erosion as they are more exposed to incoming waves (Betts, 2019). The ability to use marine shell samples in contexts where erosion has eliminated much of the terrestrial material that could be radiocarbon dated opens opportunities for even the most eroded middens to be studied, however the construction of stratigraphic chronologies with these samples may be limited if the stratigraphy has been disturbed.

The *M. arenaria* shells also display excellent agreement between intrashell measurements at the ventral margin and chondrophore. Of the three *M. arenaria* shells that were selected for intrashell ^{14}C measurements — one at the ventral margin and one at the chondrophore — they all resulted in overlapping 2-sigma calibrated probability ranges. The slight discrepancies between these measurements stems from two factors: ontogenetic growth (i.e., the ventral margin was deposited after the chondrophore) and the interplay between the ontogenetic growth of the mollusk and the ^{14}C variation in the local marine reservoir. However, the results here show that sampling the *M. arenaria* samples at the chondrophore versus the ventral margin did not result in significant differences in the calibrated probability distributions using the pre-bomb ΔR value (McNeely et al. 2006) as the calibrated ranges overlap. This further builds confidence in the use of *M. arenaria* shell for radiocarbon dating of highly eroded ghost middens. Even in cases where much of the remaining shell material is highly fragmented, these radiocarbon measurements can still be reliable if they are confirmed to be pristine aragonite.

These results are promising for the future use of *M. arenaria* samples from shell middens to study radiocarbon in Nova Scotia. However, we do acknowledge that the use of highly spatially and temporally localized ΔR values will further improve the accuracy of the marine radiocarbon measurements and will help to further study the similarities and differences between the two chronologies.

3.4.2 Improving Marine Calibrations: A Preliminary Look at Local ΔR for Port Joli Harbour

Intrashell variability is also a factor to consider when using paired marine-terrestrial samples to calculate ΔR values. The interplay between the marine radiocarbon reservoir and *M. arenaria*'s growth strategy may result in different calculated ΔR values when measurements are taken at different points on the shell, however the results here show that intrashell variability was negligible compared to the variability in calculated ΔR values from different marine-terrestrial pairs entirely. This raises some concerns about the strength of the associations between the marine and terrestrial samples and the challenges in finding secure pairs in shell middens where the stratigraphy is complex and there is a high probability of inter-layer mixing. Additionally, information on exact proximity of the marine shell samples from the previously dated terrestrial samples is not available for the samples analyzed here. This is especially a concern with samples from Area C in site AlDf-24 because of the long length of occupation and the possible inter-mixing of stratigraphic layers that represent different periods of occupation. Conversely, the rapidly deposited midden, Area A in site AlDf-24 has a much tighter range on the terrestrial dates (1522 calBP-1290 calBP) and still produces a spread in ΔR that is on the order of 250 years, similar to the spread seen from AlDf-24C samples. With only three data points for AlDf-24A, two of which are from the same shell, more data is needed to characterize the extent of ΔR variability within this midden.

A discussion of associated errors would not be complete without addressing the old wood effect: a phenomenon whereby the wood that was burned to produce the charcoal was old. The old wood effect has explained anomalous ΔR values in other studies and it has been suggested to analyze short-lived terrestrial samples rather than charcoal where possible (Kennett et al., 2002; Kim et al., 2019). Pairs 1 and 3 which have charcoal samples as the terrestrial component of the pair, have a spread of about 200 years in their ΔR calculations, and the remaining pairs used here have caribou bone and teeth as the terrestrial component. It is possible that the old wood effect plays a role in the ΔR variability, and further work could focus on only terrestrial mammal bone dates. That being said, there is just as much variability in the calculated ΔR values using charcoal as there is using caribou bone or teeth in this work.

Even though uncertainties from multiple inter- and intra- shell measurements make determining secure ΔR measurements challenging, analyzing only a single shell provides a false sense of certainty about ΔR. As outlined in detail by Martindale et al. (2018), considering only the error on a single marine-terrestrial pair tends to underestimate the uncertainty because there is no way to quantify error due to mismatched marine-terrestrial pairs. In Table 3.4, the weighted mean ΔR value is reported as 94 ± 30 years. We cautiously present this value as the first archaeological ΔR estimate for PJH. However, despite this value having a much higher geographical accuracy for the harbour compared to those values reported from the GEOSCAN survey, we recognize that its large uncertainty makes its application more challenging because it will result in the final calibrated probability ranges of marine dates much larger but should ultimately provide a more accurate calibration for the harbour. This is shown in Figure 3.11, where out *M. arenaria* dates have been recalibrated using this ΔR value.

Note the larger uncertainty ranges in Figure 3.11 compared to Figure 3.6. While this illustration is slightly circular—using marine shell radiocarbon data to calculate ΔR and then using that ΔR to calibrate the shell dates—it does show the effect that a different ΔR correction with a larger uncertainty has on the calibrated age ranges and the necessity of selecting the best ΔR value for the region in question.

There is, of course, room to improve these ΔR calculations. Some suggestions for further refinements are discussed below. First, our calculation incorporates multiple values per shell, which results in the average ΔR being skewed to represent values from these samples more closely as radiocarbon measurements are much closer within a single shell than they are between shells. For the calculation of ΔR using *M. arenaria*, it may be more favourable to use chondrophore samples only since this will avoid possible anomalies in upwelling over shorter time periods that may be captured in only a short span of ontogenetic growth at the ventral margin. The use of the ventral margin for ΔR calculations could also explain some of the variability we report.

Second, more secure pairings of marine-terrestrial samples are necessary to help build confidence in the archaeological value for the harbour. Focusing more on pairs from rapidly deposited processing middens, such as AlDf-24A may be favourable, because it lessens the severity of possible stratigraphic mixing on the ΔR calculations. While our values from AlDf-24A display a large degree of variability, the sample size is small (only three pairs from AlDf-24A) and we suspect that obtaining more measurements from other pairs in this midden will help eliminate possible outliers and provide a more robust ΔR calculation.

3.4.3 Oceanographic Contexts and Upwelling Effects on Marine Shell Radiocarbon in Port Joli Harbour

While archaeologists are challenged with choosing an appropriate ΔR value to calibrate marine dates, studying changes in this value can also provide insight into the palaeoceanographic environment of PJH. The preliminary ΔR calculations reported in this work for PJH are highly variable. This ΔR variability in the harbour has the potential to be correlated with broader oceanographic research on the Scotian shelf and modern changes in climate.

An oceanographic process that is often linked to ΔR variability is upwelling (Andrus et al., 2005; Hadden et al., 2023; Lindauer et al., 2022). Coastal upwelling is the process by which deep, ^{14}C depleted seawater is pushed to the surface thereby making the surface water appear older in terms of ^{14}C age. Upwelling usually takes on a seasonal cycle (Smith, 1992), however other mechanisms affect the ^{14}C of coastal waters, for example Sherwood et al. (2008) report a decadal scale cycle that imports ^{14}C depleted water into the Scotian shelf which is thought to be driven by the North Atlantic Oscillation. The North Atlantic is also subject to longer term oceanographic and climate cycles like the North Atlantic Oscillation which refers to changes in air pressure between the North and South Atlantic and has a significant effect on precipitation, temperature, and coastal circulation patterns (Greene & Pershing, 2003; Lotze et al., 2022).

In the North Atlantic there appears to be a more modern shift in sea surface temperature in the last few hundred years corresponding with a change in the strength of the Labrador current – an oceanographic system that carries cold water down along the

Scotian shelf – and a repositioning of the Gulf Stream, which carries warmer waters Northward (Edge et al., 2022; Keigwin et al., 2003; Lotze et al., 2022). Sediment cores show a drop in temperature prior to 1600 (AD), followed by a relatively stable period up until the last several decades where multiple proxies indicate a period of warming (Lotze et al., 2022). However, these data have yet to be extended to changes in ΔR for this region.

Sherwood et al. (2008) report a ΔR for the Scotian shelf using pre-bomb pulse gorgonian corals of known age. We have updated their data from the North East Channel with the Marine 2020 curve and the weighted mean value is -19 ± 14 years. The weighted mean from their Grand Banks samples are -7 ± 8 years (see Appendix A.4 for the updated calculations). A more recent study (Lower-Spies et al. 2020) reports a series of negative values for the Gulf of Maine extending back to 1685 AD. These values, while still negative, are mor positive than those calculated from McNeely et al. (2006) and highlight the need for spatially localized ΔR values and time-dependent considerations. Additional factors that may explain discrepancies between the pre-bomb ΔR values and the values calculated here include changes in freshwater input which may have been impacted by more modern development in the area. Studying ^{14}C at higher resolution within *M. arenaria* shells, for example within annual growth increments, could help characterize changes in upwelling. The hard-water effect, which introduces old carbon into seawater from leaching geological deposits, is also a known cause of older apparent ^{14}C ages, but bedrock geology surveys of the Port Joli area do not indicate any significant carbonate deposits (White, 2012).

There is clearly much variability in the marine reservoir values reported in the North Atlantic. Additionally, when calculating ΔR values with mollusks as we have done here, biological factors are also relevant. The ^{14}C signals from mollusks can also be impacted by their feeding strategies, which varies between species and can vary over the lifetime of a mollusk (Hadden et al., 2023; Heibert, 2015). This has warranted the suggestion that ΔR corrections may need to be species-specific (Hadden et al., 2023) Future work to understand the effects of metabolism and feeding strategies on ^{14}C measurements from *M. arenaria* will further improve the accuracy of ΔR values calculated from these species.

3.5 CONCLUSION

The complementary marine chronology for PJH shows good agreement with the terrestrial chronology reported by Betts (2019). The agreement between shell and terrestrial dates further implies that *M. arenaria* can reliably be used to date ghost middens in Nova Scotia. The radiocarbon measurements from *M. arenaria* not only displayed good agreement with their corresponding terrestrial dates, but measurements taken at the ventral margin and chondrophore of *M. arenaria* shells did not display significant differences, further building confidence in the use of this species for radiocarbon analysis in Nova Scotia. The marine chronology has some discrepancies from the terrestrial chronology, notably a possible shift in the chronology suggesting that occupation in the harbour may extend back an additional 200 years. More dates, with a more refined ΔR value for the harbour, are needed to fully characterize this shift.

We hope that the new ΔR values for PJH using the paired marine-terrestrial radiocarbon data serve as building blocks for future refinements of ΔR in PJH, thus further improving the accuracy of *M. arenaria* dates. In order to best refine this value and build confidence in a ΔR correction that can be used by archaeologists in the future, we recommend focusing more closely on rapidly deposited middens and sampling the chondrophore of *M. arenaria*. Although it has been suggested that it is best to calculate ΔR using more than one marine bivalve species (Lelièvre, 2017), more recent literature recommends focusing on one species and tailoring the ΔR to that species to account for species-specific effects of diet and metabolism on ^{14}C content (Hadden et al., 2023; Lindauer et al., 2022). The middens in PJH contain primarily *M. arenaria* shells, making the species-specific approach viable for this region and making the dating of ghost middens that contain *M. arenaria* shells more secure. In the case of *M. arenaria*, we also suggest that the chondrophore may provide a more robust ^{14}C measurement for ΔR calculations because this portion of the shell contains the average signals over the life of the clam. In contrast, sampling a portion of the ventral margin (e.g. last 2cm of growth), may increase the possibility of capturing anomalous ^{14}C spikes as a result of upwelling.

The excavations in PJH yielded an unprecedented amount of archaeological shell material, which is only beginning to be analyzed at its full potential. The meticulous work done through the E'se'get archaeology project now provides us with the means to be able to understand marine radiocarbon better in this region and to generate data that archaeologists can use to further understand the interplay between the Mi'kmaq people and their environment in southwestern Nova Scotia.

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CHAPTER 4

CONCLUSIONS

4.1 INTRA-SHELL RADIOCARBON VARIABILITY AND ΔR: FUTURE DIRECTIONS FOR COASTAL ARCHAEOLOGY IN ATLANTIC CANADA

The complexities of marine radiocarbon data are worth confronting as marine shells are found more frequently than any other samples that are viable for radiocarbon analysis in coastal archaeology sites (Hadden et al., 2023). Despite the complex interplay between mollusk shell growth strategies, time-averaging effects, and time-variable local marine reservoir offsets (ΔR), archaeologists can build confidence in their marine shell dates for one-off chronometric dating purposes by assessing the uncertainties associated with both intra-shell radiocarbon variability and local ΔR values. This requires incorporating aspects of sclerochronology, thorough data analysis, and continuing to study sites with both marine and terrestrial radiocarbon samples to calculate spatially localized ΔR values.

Intra-shell radiocarbon variability is often used to study past changes in climate and oceanography, but this variability creates challenges for using marine shell for one-off chronometric dates in archaeological contexts (Butler et al., 2009; Jones et al., 2010; Scourse et al., 2006, Culleton et al. 2006). Although high-resolution intra-shell radiocarbon measurements may not be necessary for every project, simply obtaining a few radiocarbon dates per shell can help to ensure that samples with the least amount of variability are chosen for archaeological applications (Hadden et al. 2017). The R-Combine function in Oxcal is an accessible method for archaeologists to analyze multiple dates from the same shell. *C. virginica* from P.E.I. returned intrashell measurements with the best agreement. This is likely a factor of the individuals studied being short-lived, the species' growth strategies, and the marine environment in P.E.I. Both coastal Nova Scotia and British Columbia are influenced by current systems that cause seasonal to decadal cycles in ocean circulation that impact the dissolved inorganic carbon in these respective regions.

Further applying sclerochronological methods of growth line analysis can also provide a more exact number for how many years of ontogenetic growth are averaged over in any given radiocarbon measurement. The samples I investigated in Chapter 2 — *Crassostrea virginica* from Prince Edward Island, *Mya arenaria* from Nova Scotia, and *Saxidomus gigantea* from British Columbia — each have different growth strategies, resulting in different time-averaging effects for the radiocarbon measurements taken throughout their shells. Applying sclerochronological methods requires a knowledge of growth patterns specific to each species being studied and the three species considered in this case study are only a subset of an unknown large number of species that have been used in archaeological applications. Questions of intra-shell variability and ontogenetic growth must be considered on a case-to-case basis in archaeological applications.

While species-specific considerations are crucial for building confidence in marine shell dates, these studies should also be complemented with site-specific case studies such as the one I present in Chapter 3 from Port Joli Harbour. The choice of an appropriate ΔR value is also crucial for the accurate calibration of marine shell radiocarbon dates. The series of marine dates from *M. arenaria* shells reported for Port

Joli Harbour, Nova Scotia showed good agreement with the existing terrestrial chronology even when calibrated with a modern ΔR value. However, this type of comparative analysis between marine and terrestrial radiocarbon data is not possible in midden sites that are highly eroded and contain little to no terrestrial samples (M. Betts, 2022). To ensure secure dates on stand-alone marine samples, ΔR values should continue to be refined.

Using the paired marine-terrestrial samples, ΔR spatially localized to Port Joli harbour were calculated and were highly variable. These values were consistently more positive than the modern value from the online database and from values reported in oceanographic literature for the Scotian Shelf (McNeely et al., 2006; Reimer, n.d.; Sherwood et al., 2008). This archaeological value should be further refined by analyzing more marine-terrestrial pairs, perhaps avoiding charcoal to mitigate the old-wood effect and conducting more rigorous statistical analyses that eliminate outliers in the data (Cook et al., 2015; Martindale et al., 2018). Refining local ΔR values using data from coastal sites like those within Port Joli harbour that have both terrestrial and marine samples should be of crucial importance to coastal archaeologists. As ΔR values are further refined for the harbour, marine shell radiocarbon dates will become even more accurate and an even more viable option for studying highly eroded "ghost middens" that contain no terrestrial samples and ensure they are not overlooked (M. Betts, 2022; M. W. Betts, 2019).

4.2 THE FEMINIST APPROACH TO RESEARCH REVISITED

Adopting a feminist approach to research provides a framework for questioning long-held assumptions about the importance of time and chronology in archaeology and calls for a degree of data transparency that is not currently the norm in archaeology, but that is necessary to reinforce realistic interpretations of marine radiocarbon data. This approach also encourages rigorous questioning of data and a critical analysis of all the uncertainties involved in radiocarbon data. Throughout this thesis, a high priority has been placed on communicating uncertainties in each step of the process from sampling strategies to calibration to archaeological interpretations with the hope that others will do the same in future studies.

The feminist literature in archaeology emphasizes its reactionary framework, calling into question current norms and biases in how archaeology is conducted and who conducts it (Kincaid et al., 2007; Wylie, 2007; Wylie & Hankinson Nelson, 2007). The work outlined in this thesis is situated as a reaction against the black box approach. Because radiocarbon from marine mollusk shells cannot be treated the same as terrestrial samples and it requires an interdisciplinary approach archaeologists have to be comfortable with adopting and developing different standards for analysis. For example, perhaps it will become standard to obtain multiple radiocarbon samples per shell rather than one and perhaps more archaeologists will learn and adopt methods of material characterization to understand the quality of their samples. If archaeologists want to continue to build confidence in marine shell radiocarbon dates, they must continue to deeply question their own samples.

The feminist approach to research is also a reaction against the culture-historical framework and how radiocarbon data is typically used to uphold this framework. While radiocarbon chronologies are extremely valuable sources of information for archaeologists, chronologies are not perfect ladders that represent concrete steps toward the modern day, but they are messy, unstable, ever-changing sequences of probabilities that may change as calibration curves are updated, more dates are included, and ΔR values are refined. For example, although the marine dates from Port Joli show agreement with the terrestrial chronology from, the marine dates display larger uncertainties in the calibrated ranges and these values may change with updated ΔR calculations.

Archaeologists must be willing to continuously question previous assumptions, data, analysis, and interpretations to continue to build confidence in marine shell radiocarbon in Canadian archaeology. While the "radiocarbon revolutions" in archaeology have made the calibration and analysis of dates very accessible (Bayliss, 2009), the approach to the analysis done prior to obtaining dates and the interpretations done after the dates have been calibrated may also shift in the future. As highlighted by other recent works (Hadden et al., 2023; Lindauer et al., 2022), building confidence in marine shell radiocarbon requires interdisciplinary collaboration between materials science to characterize diagenesis, oceanography to understand changes in the marine environment, and biology and sclerochronology to study growth strategies will make these radiocarbon measurements more robust and archaeological interpretations stronger.

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APPENDIX A

A.1 FT-IR BACKGROUND AND ADDITIONAL DATA FOR CHAPTER 2

Transmission Fourier Transform Infrared Spectroscopy probes the vibrational modes within a sample by analyzing how the sample interacts with infrared radiation. Depending on the chemical and structural properties of the material, different frequencies of infrared radiation will be absorbed by the sample and will appear as peaks in the FTIR spectrum. Despite having the same chemical formula, calcite and aragonite have different absorption peaks in the mid IR range due to their different crystal structure. This makes FTIR a good candidate for detecting diagenesis in the form of the dissolution and reprecipitation of aragonite and calcite (Weiner, 2010). Re-precipitated calcite from groundwater in the burial environment of the aragonitic marine samples can incorporate older or younger carbon from the marine environment, resulting in radiocarbon dates that do not accurately reflect the age of the mollusk shell. This detection method works specifically well for shells that mineralize only aragonite, since the presence of any calcite in archaeological samples would indicate diagenesis. For *M. arenaria* and *S. gigantea*, this method works well because both shells are composed entirely of aragonite. However, for the Atlantic Oyster, *C. virginica*, which mineralizes both calcite and aragonite into the shell, this approach is less insightful.

Attenuated total reflectance (ATR) FT-IR was used to analyze the carbonate from each of the samples. Samples were manually cleaned with water and allowed to dry fully. Samples were collected by scratching off powder from both the inner and outer portions of each of the shell sampling locations. All samples were analyzed using the Bruker

Alpha II spectrometer with the platinum ATR attachment, and peak positions for the spectra were obtained using the single peak pick function in the OPUS software.

Example spectra of calcite and aragonite are shown in Figure A1.1. The Greek letter ν is used to denote specific vibrational frequencies within the calcium carbonate lattice. The ν_3 corresponds to asymmetric stretching of the CO_3^{2-} moiety, the ν_2 peak corresponds to out-of-plane bending, the ν_4 peak to in-plane bending, and the ν_1 peak to symmetric stretching (White, 1974). The ν_4 peak is split into two sub-peaks in the aragonite spectrum, denoted as ν_{4a} and ν_{4b}. A combination mode of both the symmetric stretching (ν_1) and in-plane bending (ν_4) is present and is denoted as $\nu_1 + \nu_4$.

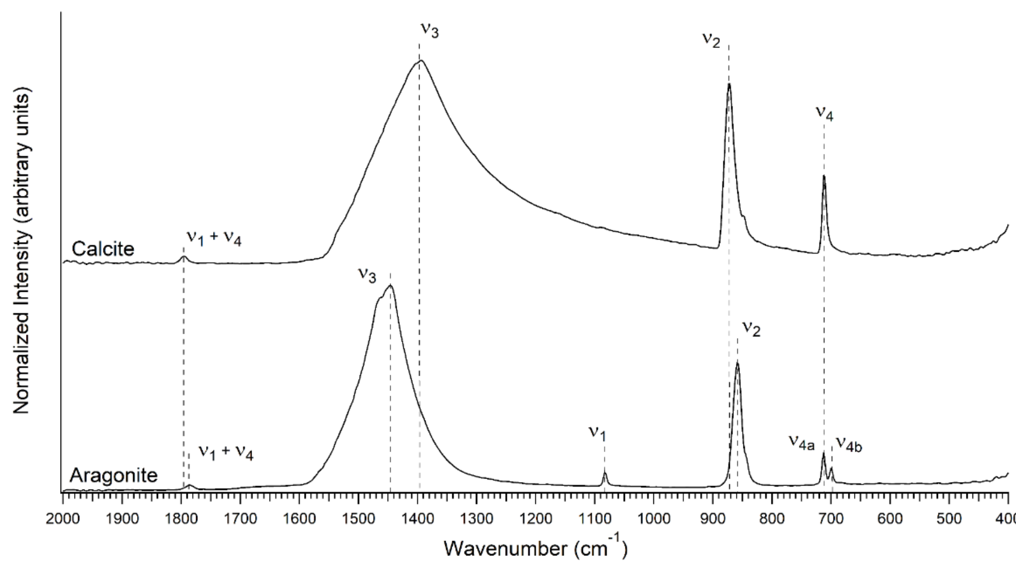

Figure A1.1 ATR FT-IR spectra of calcite (top) and aragonite (bottom) in the mid-IR region with characteristic peaks shown.

For each spectra shown below, the expected positions of calcite peaks are shown as blue dotted lines and are labeled on each plot to compare to the measured peaks. In ATR FT-IR, the sample is crushed into a fine powder using a mortar and pestle and is pressed against the surface of a platinum crystal for analysis. The infrared beam is directed into the crystal and reflected back and forth, allowing the beam to interact with the sample powder. The sample will absorb certain frequencies of the infrared beam according to the vibrational modes of the sample. These absorbed vibrational modes appear as peaks in the resulting spectra. By convention the frequencies are reported as wavenumbers, in units of cm^{-1}, which is directly proportional to the energy of the vibrational modes. The spectra were obtained using a Bruker Alpha II spectrometer with 24 scans and a resolution of 4 cm^{-1} and have an uncertainty of +/- 2 cm^{-1}. The expected peaks for calcite were determined by analyzing purchased Alfa Aesar calcium carbonate powder (spectrum of this sample shown in Figure 2.2 in Chapter 2).

A.1.1 *Crassostrea virginica*

IR spectra from the oyster samples were consistent with a mixture of calcite and aragonite. The secondary shoulders on the ν_2 peak toward lower wavenumber is indicative of aragonite, and the ν_1 peak which is only present in aragonite is detectable in the samples from *C. virginica*. The sample taken at the hinge of shell 3488 also have a secondary ν_4 peak which is consistent with aragonite. Spectra from shell sample 3488 are shown in Figure A1.2, where the shoulders on the peaks are visible, indicating the presence of aragonite.

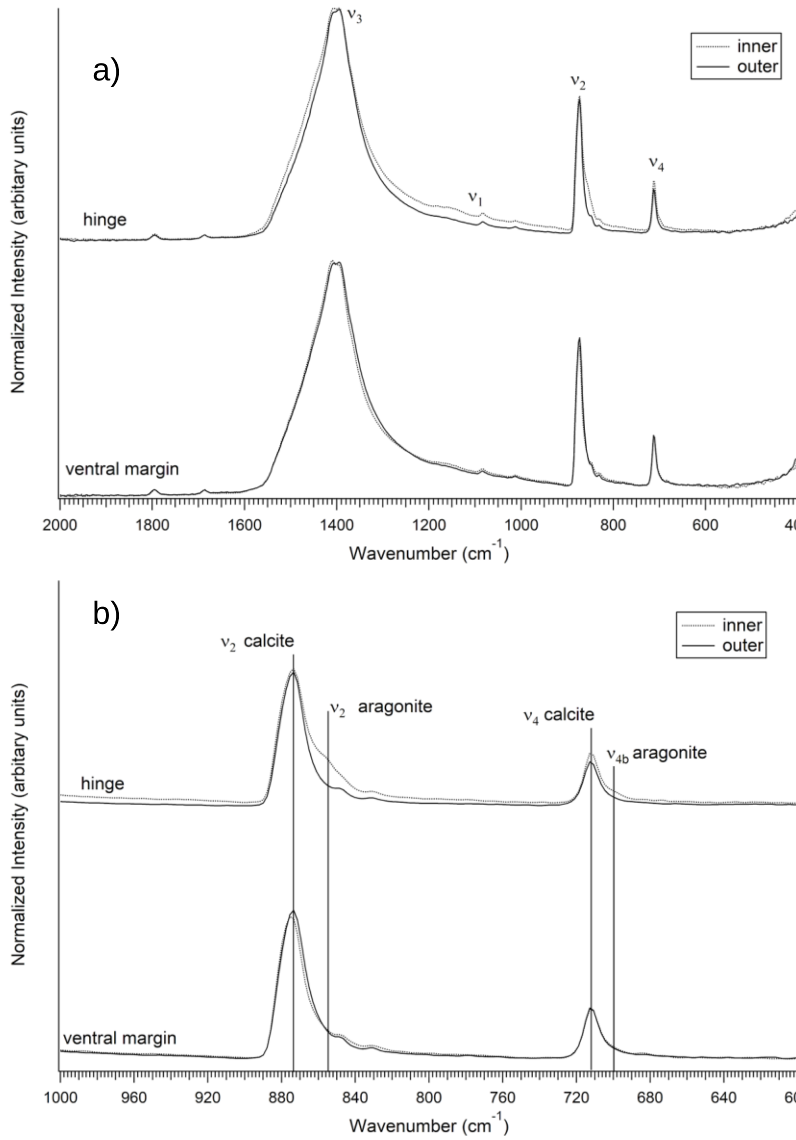

Figure A1.2 FT-IR spectra from oyster shell 3488. Dotted lines indicate spectra taken from the outer surface of the shell and solid lines from the inner portion. Figure a) shows the entire fingerprint region for calcium carbonates and b) shows only the ν_2 and ν_4 peaks.

This mixture that we report here is consistent with the expected mineralogy of oysters. Particularly, the innermost layer of *C. virginica* is aragonite, which forms the lustrous nacre, as well as muscular components, the remaining carbonate is calcite (Marin et al., 2012). *C. virginica* has also been reported to form alternating layers of calcite and aragonite deposition in the hinge portion of the shell (Carriker & Palmer, 1979).

The shoulder on the ν_2 peak at 848 cm^{-1} has been reported previously for calcite and has been investigated in relation to isotopic substitutions of ^{12}C for ^{13}C (Xu et al., 2018). The shoulder on the ν_3 peak toward lower wavenumber is also characteristic of calcite (Xu et al., 2018). Subtle peaks at 1013 and 1687 cm^{-1} were detectable in nearly all the oyster samples and may be attributed to traces of clay but are likely not linked to the structure of the carbonate.

Additional IR spectra from the oyster samples 2608 and 2982 are shown in Figure A1.3. As with sample 3488, these appear to be primarily calcite with trace aragonite.

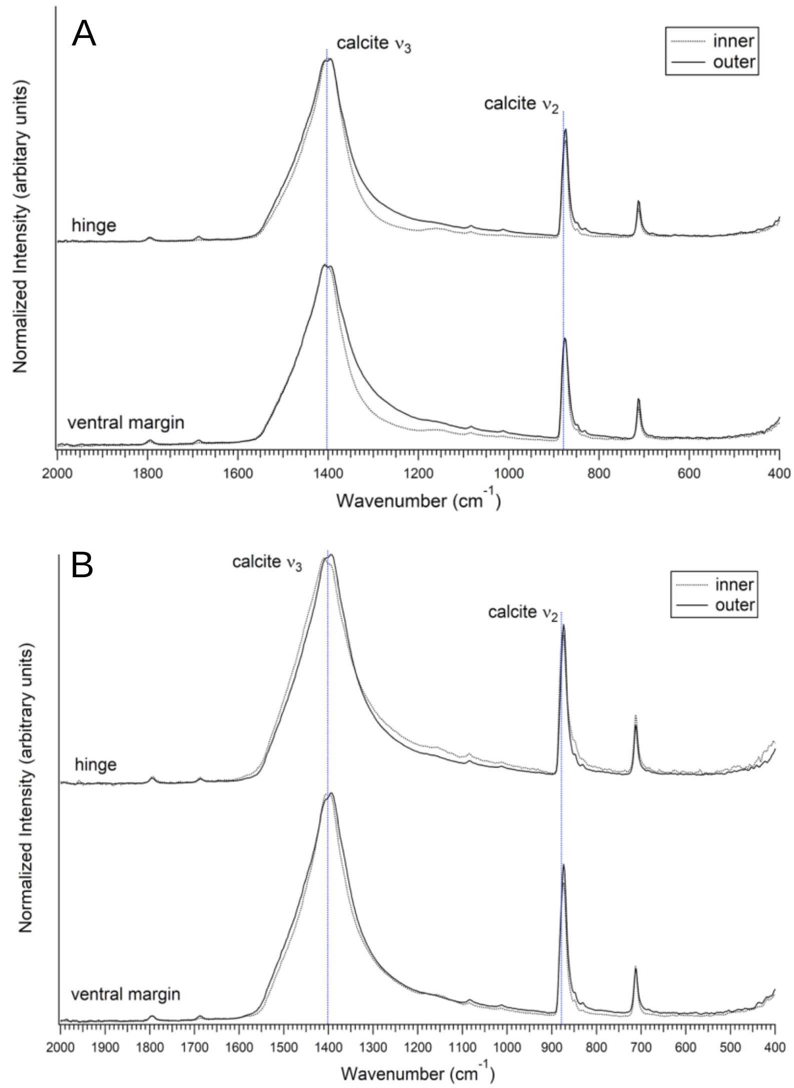

Figure A1.3 Additional ATR FT-IR spectra of oyster samples 2608 (A) and 2982 (B). Samples taken from the inner portion of the shell are denoted by the dotted line and samples taken from the outer portion are denoted by the solid line.

A.1.2 *Saxidomus gigantea*

The ATR FT-IR spectra collected on the samples of *S. gigantea* from Comox and Deep Bay, British Columbia are shown in Figures A1.4, A1.5, and A1.6. All samples are consistent with aragonite, indicating no diagenesis in the form of calcite reprecipitation.

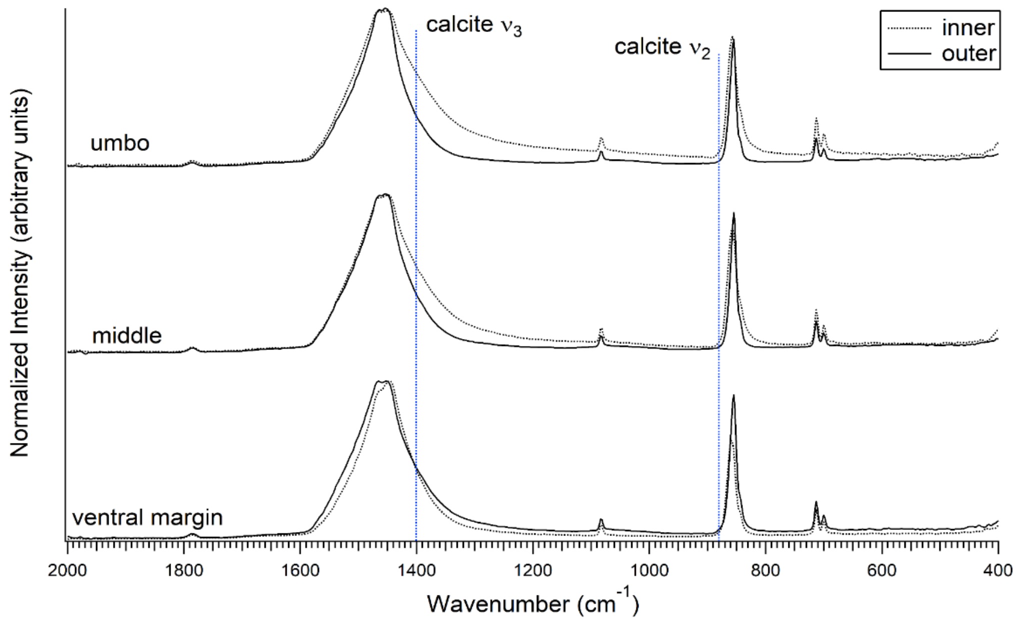

Figure A1.4 ATR FT-IR spectra from sample 7039, from site DiSe-7 (Deep Bay, British Columbia). Dotted lines indicate samples taken from the inner surface of the shell and solid lines indicate samples taken from the outer surface of the shell.

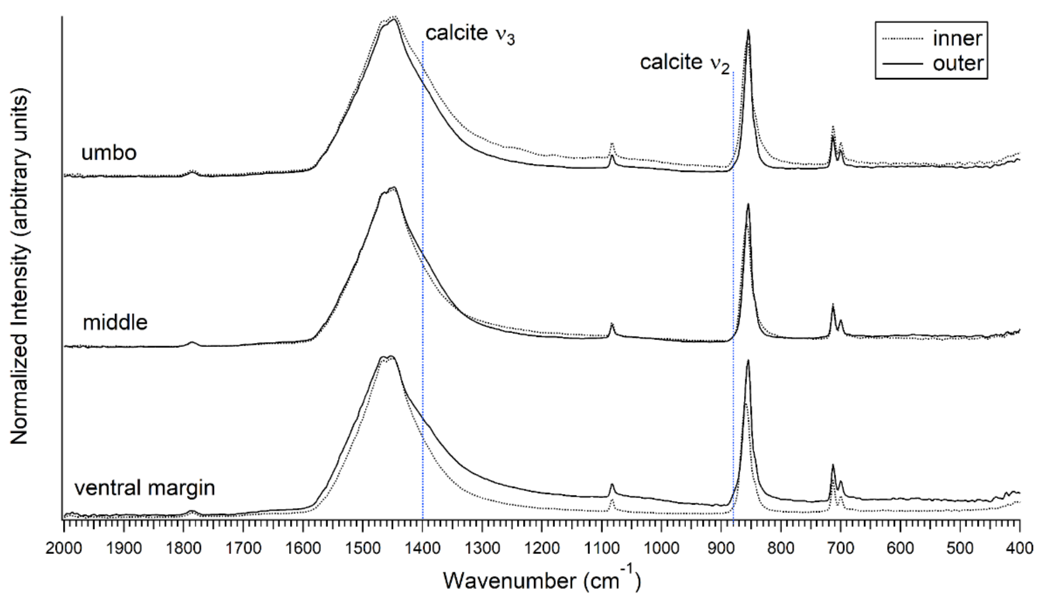

Figure A1.5 ATR FT-IR spectra from sample 7041, from site DiSe-7 (Deep Bay, British Columbia). Dotted lines indicate samples taken from the inner surface of the shell and solid lines indicate samples taken from the outer surface of the shell.

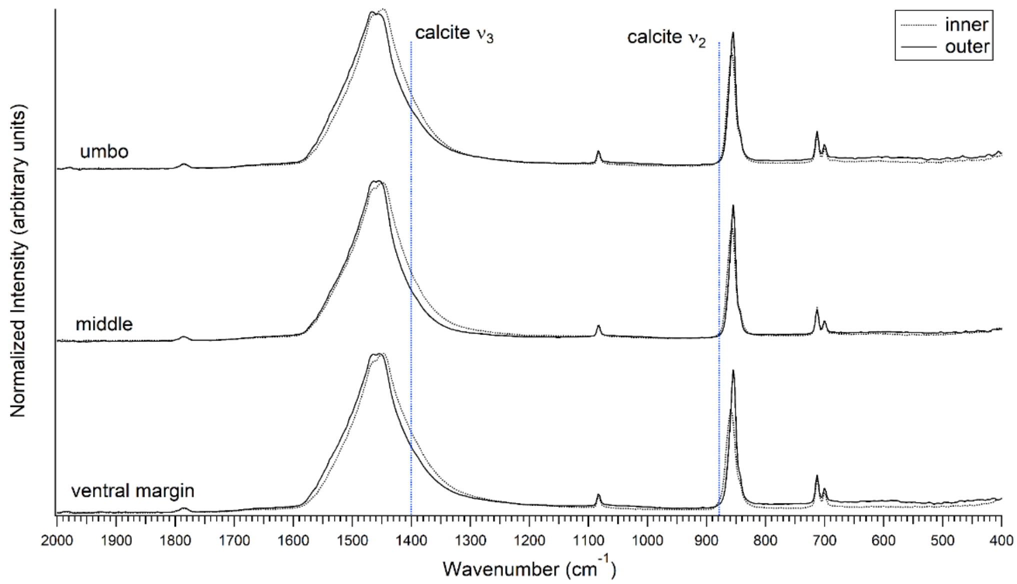

Figure A1.6 ATR FT-IR spectra from sample 7050, from site DkSf-20 (Comox, British Columbia). Dotted lines indicate samples taken from the inner surface of the shell and solid lines indicate samples taken from the outer surface of the shell.

A.1.3 *Mya arenaria*

The ATR FT-IR spectra on *M. arenaria* from Port Joli, Nova Scotia are shown in Figures A1.7, A1.8, and A1.9 and the peak positions are reported in Table A2. All samples are consistent with aragonite, indicating no diagenesis in the form of calcite reprecipitation.

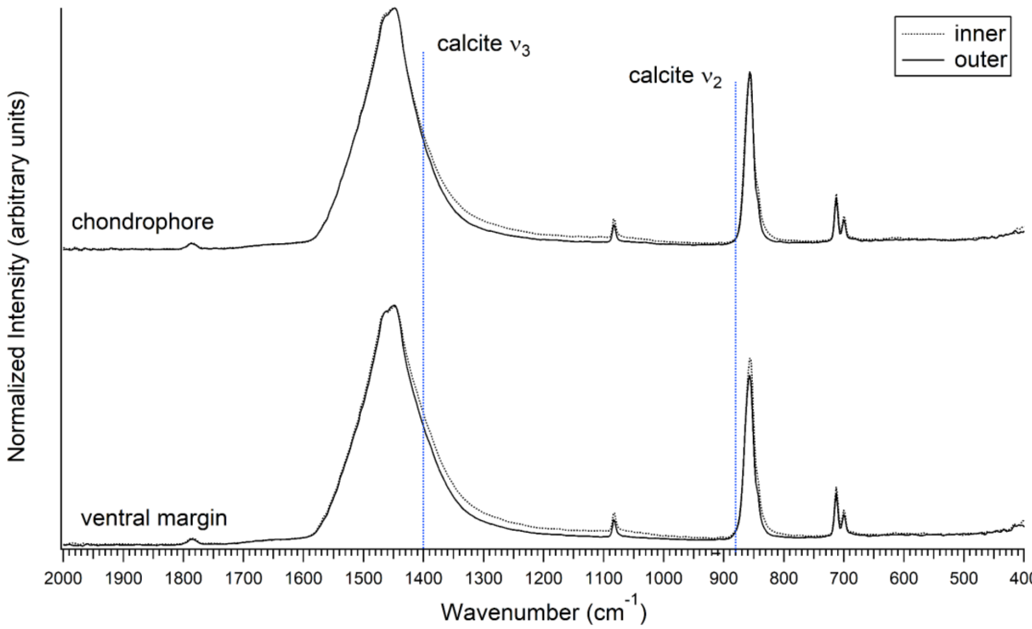

Figure A1.7 ATR FT-IR spectra from *M. arenaria* sample 1 from Port Joli, NS. Dotted lines denote samples taken from the inner surface of the shell and solid lines denote samples taken from the outer surface of the shell.

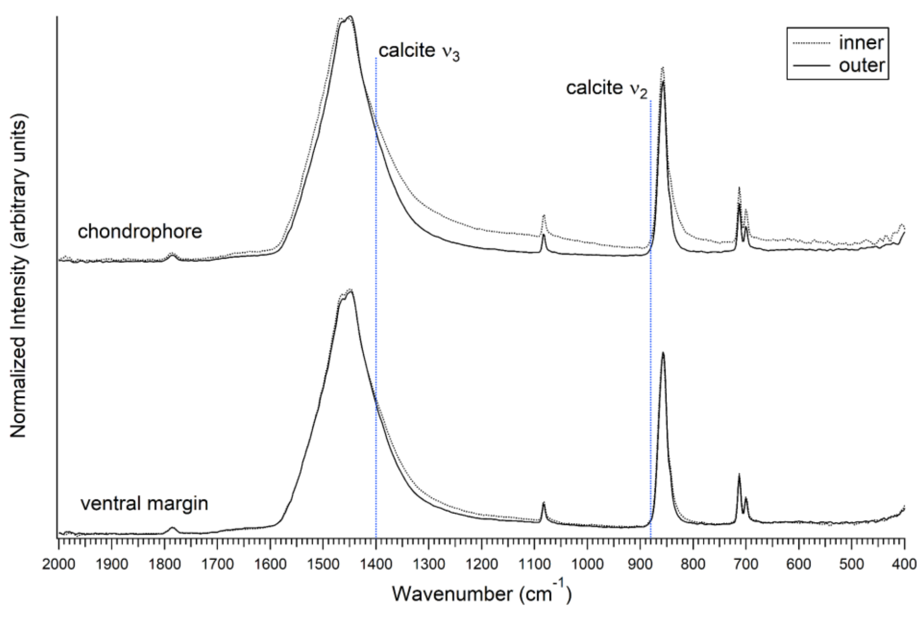

Figure A1.8 ATR FT-IR spectra from *M. arenaria* sample 8c from Port Joli, NS. Dotted lines denote samples taken from the inner surface of the shell and solid lines denote samples taken from the outer surface of the shell.

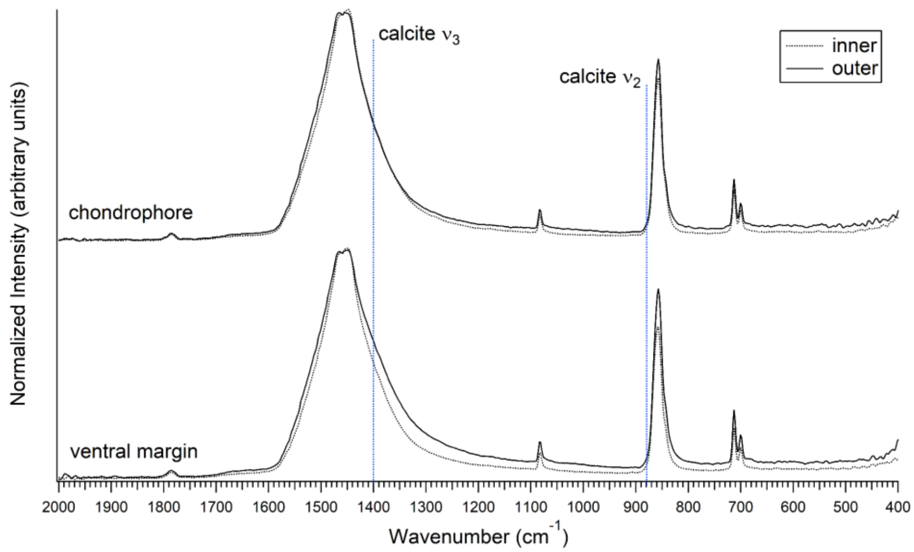

Figure A1.9 ATR FT-IR spectra from *M. arenaria* sample 9b from Port Joli, NS. Dotted lines denote samples taken from the inner surface of the shell and solid lines denote samples taken from the outer surface of the shell.

A.2 ADDITIONAL FT-IR DATA FOR CHAPTER 3

FT-IR spectra of samples of the ventral margin of *M. arenaria* analyzed for radiocarbon in Chapter 3 are displayed in Figure A2.1. All ventral margin samples were analyzed using the Transmission attachment for the Bruker Alpha II spectrometer. In transmission FT-IR, the sample is crushed into a powder using a mortar and pestle and is then mixed with potassium bromide (KBr) powder and is pressed into a small pellet using a small hydraulic press. The pellet is placed into the sample chamber and the infrared beam passes directly through the sample pellet. As with ATR FT-IR, the frequencies that are absorbed by the sample appear as peaks in the resulting spectra. The KBr has no effect on the final spectra as it has no active vibrational modes in the infrared region, it is simply used as a medium to evenly disperse the sample powder. The spectra were obtained using a Bruker Alpha II spectrometer with 24 scans and a resolution of 4 cm^{-1}.

Peak shapes and thus positions for aragonite shift slightly between the transmission collection method and the ATR method, but the peaks represent the same vibrational modes (Khoshhesab, 2012). All samples were found to be aragonite, with no discernable traces of calcite. The expected peak positions for calcite which are labeled in Figure A2.1 are from Weiner (2010).

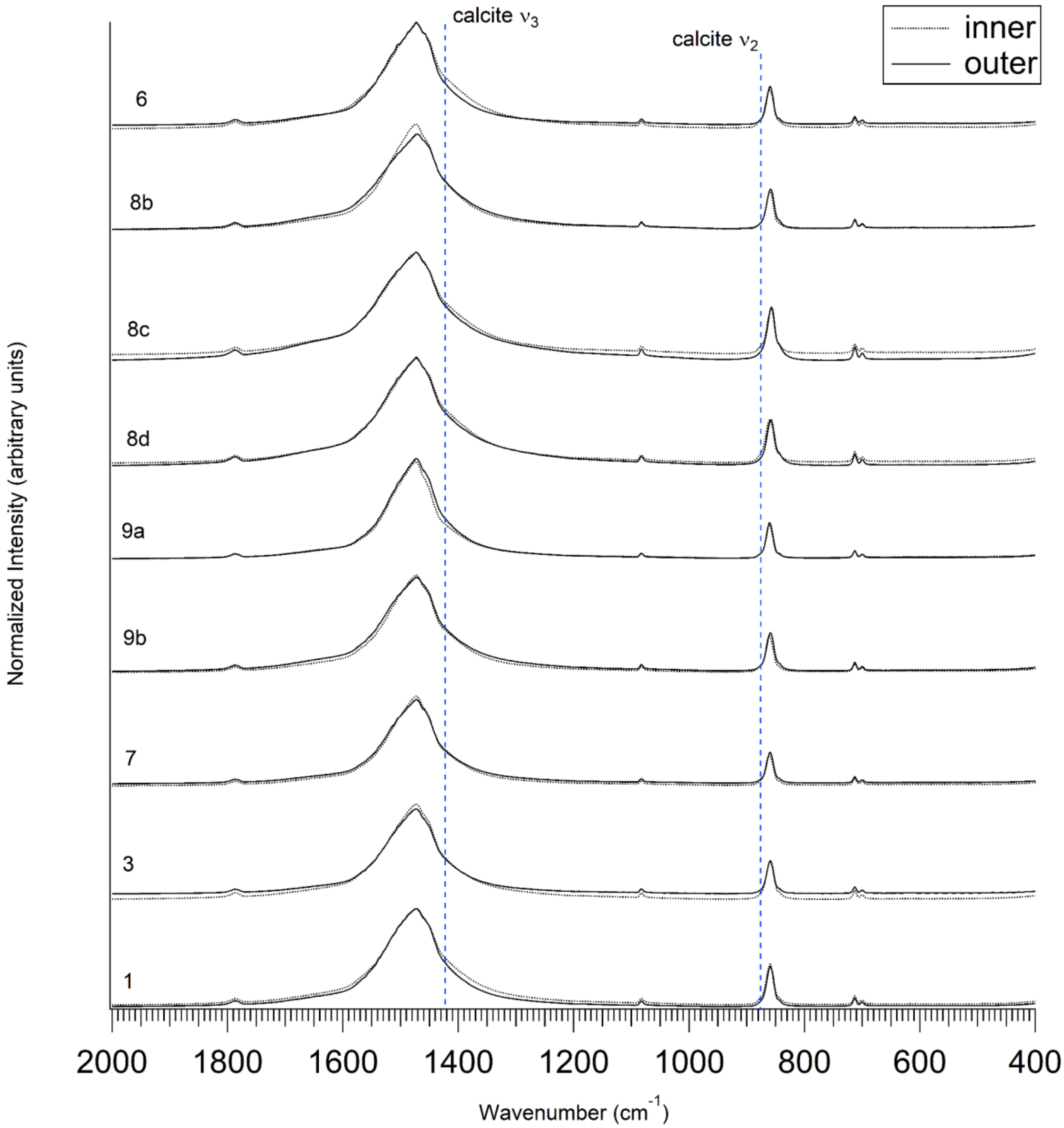

Figure A2.1 Transmission FT-IR spectra of the inner and outer surfaces of the ventral margin of the *M. arenaria* samples analyzed for radiocarbon in Chapter 3. The peaks are consistent with aragonite.

APPENDIX B

B.1 MCNEELY ET AL. (2006) DATA FROM THE SCOTIAN SHELF

The raw data from the online ΔR database (Reimer & Reimer 2001) is reported below in Table B.1. These data were calculated from historic live-collected samples from 1908-1905. The distance in kilometers from Port Joli Harbour is also reported. The weighted mean can be calculated automatically on the database.

Table B.1: The five closest ΔR values to Port Joli Harbour from the online database (Reimer & Reimer 2001).

Locality	**Collection Year**	**Distance from Port Joli (km)**	**ΔR**	**Error**
Lockeport, NS	1950	26	-53	60
LaHave Island, NS	1910	59	-47	50
Barrington Passage, NS	1910	72	-47	50
Barrington Passage, NS	1910	72	-72	25
Black Rock, NS	1908	76	-148	30
Weighted Mean			-88	45

B.2 UPDATED PRE-BOMB ΔR VALUES FROM SHERWOOD ET AL. (2008)

The values for the collection year and marine 14C of gorgonian corals are reproduced from Table 1 in Sherwood et al. (2008). These values were input into the online ΔR calculator, and the resulting values are reported (Reimer & Reimer 2001). The weighted mean values were calculated following Martindale et al. (2018). Only values from the North East Channel and Grand Banks are presented because of their proximity to Port Joli Harbour.

Table B.2: Gorgonian coral data from the North East Channel and Grand Banks reproduced from Table 1 in Sherwood et al. (2008). Values are updated to the Marine2020 calibration curve. (Heaton et al., 2020, Reimer & Reimer 2001).

North East Channel	**Collection Year**	**Marine 14C age**	**ΔR**	**2σ**	**1σ**
	1947	630(35)	27	71	36
	1948	620(30)	17	60	30
	1942	545(30)	-59	60	30
	1934	595(30)	-9	60	30
	1924	550(30)	-55	60	30
Weighted Mean			-19		14
Grand Banks					
	1943	570(25)	-34	51	26
	1934	605(30)	1	60	30
	1925	585(25)	-20	51	26
	1919	590(25)	-15	51	26
	1910	675(25)	68	51	26
	1896	575(25)	-43	51	26
	1884	625(30)	-5	60	30
Weighted Mean			-7		8

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