Radiocarbon, Vol 56, Nr 1, 2014, p 305–311 DOI: 10.2458/56.16509

RADIOCARBON VARIABILITY IN *CRASSOSTREA VIRGINICA* SHELLS FROM THE CHESAPEAKE BAY, USA

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ABSTRACT. Fifteen accelerator mass spectrometry (AMS) radiocarbon dates obtained on small subsections of archaeological and historical *Crassostrea virginica* (eastern oyster) shells provide a means to test for intrashell variability in ^{14}C content in late Holocene Chesapeake Bay mollusks. Although salinity and temperature vary considerably throughout the year, the Chesapeake Bay generally lacks the strong coastal upwelling present in the eastern Pacific where intrashell ^{14}C variability is significant. Intrashell variability in Chesapeake Bay *C. virginica* is between ~60–100 ^{14}C yr, considerably smaller than the 120–530 ^{14}C yr ranges noted for shells from strong upwelling zones. As a precaution, we follow Culleton et al. (2006) and argue that large subsamples of shells across multiple growth increments are ideal for AMS ^{14}C dating of mollusks to offset potential issues of intrashell ^{14}C variability.

INTRODUCTION

In coastal and other aquatic regions, radiocarbon dating of mollusk shell is common, but marine samples require reservoir corrections (ΔR) that may vary significantly across space and through time (see Stuiver et al. 1986; Southon et al. 1990; Kennett et al. 1997, 2002; Culleton 2006; Petchey et al. 2008). Accelerator mass spectrometry (AMS) ^{14}C dating of small subsamples from annual growth bands or other short growth intervals on mollusk shells may also result in intrashell differences in ^{14}C ages, especially in areas like California and Peru that have strong seasonal upwelling where marine currents can bring old carbon to the surface at varying seasonal or other cyclical intensities (Culleton et al. 2006; Jones et al. 2007). Such intrashell variation can result in differences in ΔR as great as 120–530 yr on a single shell, posing challenges for researchers working to develop high-precision chronologies (Culleton et al. 2006; Jones et al. 2010). Five ^{14}C dates recently reported from a single archaeological *Mercenaria mercenaria* (hardshell clam) shell from St. Catherine's Island, Georgia (USA), however, indicated that the effects of seasonal upwelling or marine circulation were limited (~80 ^{14}C yr or less) along this portion of North America's Atlantic coast (Kennett and Culleton 2012; Thomas et al. 2013). These data raise questions about intrashell ^{14}C variability in other mollusk species and geographic locations.

This article presents AMS ^{14}C dates obtained on *Crassostrea virginica* (eastern oyster) shells from Chesapeake Bay, USA, to understand the intrashell ^{14}C variability of mollusks from a large estuary (Figure 1). *C. virginica* is found throughout North America's Atlantic coast and Gulf of Mexico and is commonly used for ^{14}C dating in areas with established ΔR (Colman et al. 2002; Thomas 2008; Rick et al. 2012; Thomas et al. 2013). The Chesapeake Bay, similar to other North American Atlantic coast estuaries, generally does not have the intense upwelling found on the Pacific coast, suggesting that intrashell ^{14}C variability may not be as pronounced in this region. However, previous research has demonstrated subregional variability in ΔR for the Chesapeake Bay, with larger values for the western shore and generally lower values for the eastern shore that are likely influenced by ^{14}C-depleted freshwater that enters the bay from some if its drainages, ^{14}C-depleted seawater that enters the bay at its mouth, and/or biological carbon recycling (Rick et al. 2012). This study builds on this work by investigating variability in ΔR in individual mollusk shell growth bands for the Chesapeake Bay, focusing primarily on how these patterns may affect archaeological and geological ^{14}C chronology building in the region and along the broader North American Middle Atlantic coast.

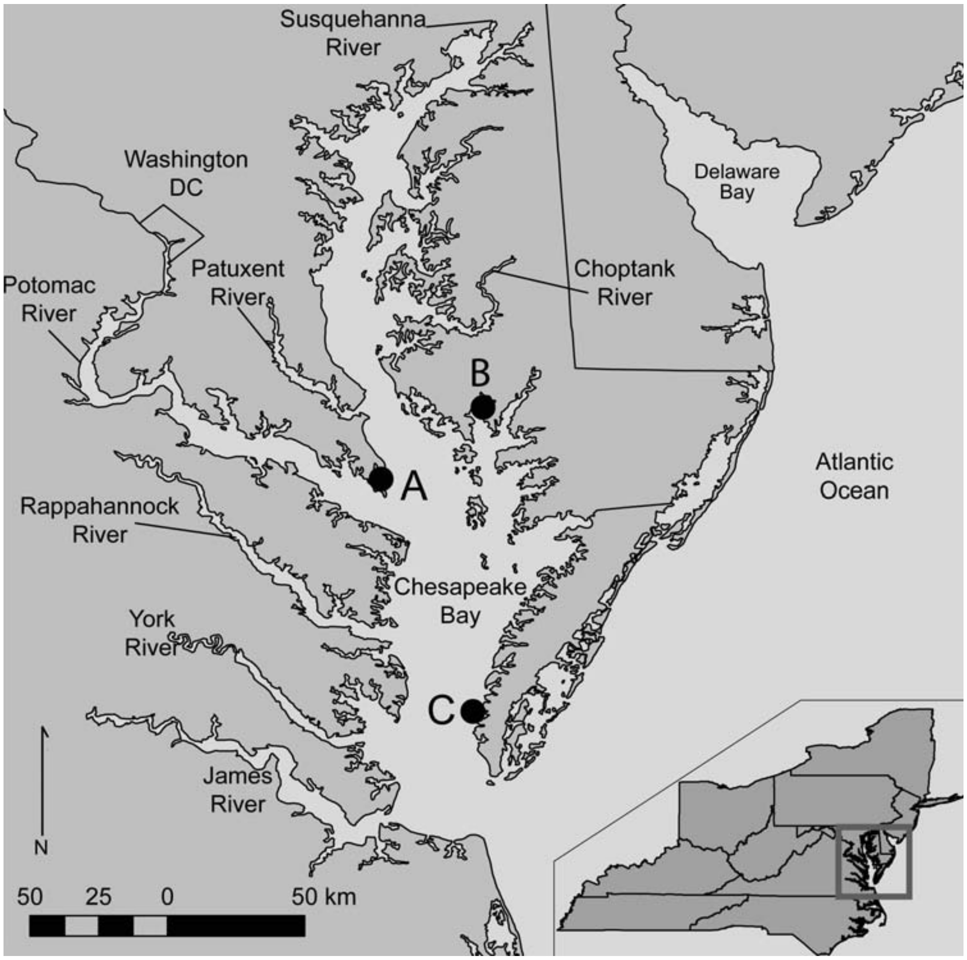

Figure 1 Chesapeake Bay showing locations of historical and archaeological mollusk shells analyzed in this study: A) Point Lookout, Maryland (OS-95242, -95243, Beta-284384); B) Fishing Bay, Maryland (OS-94972, -95091, -95092, -95093, -95094, -95095, -95096, -95097, -101201); C) Savage Neck, Virginia (OS-95244, -95249, -101203). The inset shows the mid-Atlantic Bight region and other eastern states of the USA.

MATERIALS AND METHODS

We selected four complete *C. virginica* shells (left valves) for AMS analysis, including one historical and three archaeological specimens. We obtained a total of 15 ^{14}C dates with between 3–5 samples from each valve. The historical oyster shell was collected in 1898 near Point Lookout, Maryland, at the mouth of the Potomac River. Three samples were analyzed from this specimen, including one previously reported sample (Rick et al. 2012). The 12 archaeological samples were obtained from three valves from three archaeological sites. Two of these specimens come from Fishing Bay, a Chesapeake subestuary on Maryland's eastern shore. Four samples were obtained from one valve collected at the Middle Woodland (2450–1050 ^{14}C BP) site of 18DO130 and five samples were obtained from one valve from the Late Woodland (1050–350 ^{14}C BP) site of 18DO439 (Rick et al. 2011). Three samples were analyzed from an oyster valve collected during excavation at 44NH478, an Early Woodland (3150–2450 ^{14}C BP) shell midden located on Virginia's eastern shore. These samples provide good coverage of the bay across a ~3000-yr period, from the Potomac River to near the mouth of the Chesapeake.

All oyster shells were physically cleaned using ultrasonication, an abrasive brush, and deionized water. Prior to subsampling, the outermost shell surface (the area most susceptible to contamination) was removed using a Dremel® rotary drill and a diamond burr. Small subsections of the shells were then milled parallel to the shell growth axis and collected in glass vials. We subsampled at a variety of intervals across the shells to capture potential variability within an individual shell. Our sampling intervals range from 2 to 20 mm apart and were dictated by total shell length and the overall preservation of the oyster shells (Tables 1 and 2). Samples DO130-1 and DO130-4 were obtained from roughly the same distance from the umbo (72 mm), but at different points about 2 mm apart along the axis perpendicular to the umbo.

Oysters can live up to 20 yr (Buroker 1983), but most Chesapeake archaeological specimens are much younger (~4–6 yr; Waselkov 1982:200; Gibb and Hines 1997). Oyster growth is influenced by the composition of the oyster reef, substrate, and other factors that result in a variety of shapes and sizes of individual shells. *C. virginica* is also subject to fouling by a variety of organisms that can affect shell preservation and quality, which is not always visible on the surface. We avoided areas with alteration from polychaete worms and *Cliona* spp. (boring sponges) that could result in localized shell recrystallization.

Fourteen samples were sent to the National Ocean Sciences AMS (NOSAMS) Facility at the Woods Hole Oceanographic Institution. One previously reported sample (Beta-284384) was analyzed by Beta Analytic, Inc. (Rick et al. 2012). All shell subsamples were etched in a dilute HCl bath to remove labile carbonate. Details of the AMS ^{14}C dating procedures are provided online at http://www.whoi.edu/nosams (NOSAMS) and http://www.radiocarbon.com (Beta Analytic).

For the 3 known-age specimens from Point Lookout, Maryland (USA), we calculated marine ΔR, the difference between the local and global marine reservoir (Stuiver et al. 1986). Marine model ages were obtained from the Marine09 calibration data set (Reimer et al. 2009). To calculate ΔR, we relied on the following equation:

$$\Delta \mathrm{R} = P - Q$$

where P is the measured ^{14}C age of a sample of known age and Q is the marine model age obtained from the Marine09 calibration curve (Stuiver et al. 1986). Uncertainty in ΔR was calculated as $\Delta R = (P^2 + Q^2)$ (Stuiver et al. 1986). Weighted averages of ΔR for the 3 Potomac River samples were calculated by using the greater value of the weighted uncertainty or the standard deviation as the weighted average error (Stuiver et al. 1986; Culleton 2006; Jones et al. 2007). We use a chi-squared (χ^2) test to evaluate if the distribution of the ΔR values and other ^{14}C dates was greater than statistically expected (Ward and Wilson 1978).

For the 12 archaeological samples, we calibrated all of the dates using the OxCal v 4.2 calibration program (Bronk Ramsey 2009) and the Marine09 calibration curve (Reimer et al. 2009). We applied a ΔR of –88 ± 23 yr for the dates from 18DO130 and 18DO439 and ΔR of 2 ± 46 yr to the dates from 44NH478 (Rick et al. 2012).

RESULTS

The three dates on the historical *C. virginica* from Point Lookout, Maryland, yielded ΔR values that ranged from 44 ± 34 to 144 ± 46 yr, a difference of 100 yr (Table 1). The value of 144 ± 46 yr was taken from the distal end of the shell and was a subsample that crossed over multiple shell growth bands. In contrast, the lower values were taken from 41 and 44 mm from the shell umbo and sampled a more limited range of shell growth. When all 3 samples are averaged together, the average

ΔR is 81 ± 29 yr ($T = 3.07$, $\chi^2_{0.05} = 5.99$), a value near the lower range of previously reported values (82 ± 46 to 148 ± 46 yr) for the Chesapeake Bay's western shore (Rick et al. 2012).

Table 1 Radiocarbon and reservoir model data from a single historical *Crassostrea virginica* shell from Point Lookout, Maryland, collected in 1898.

Lab ID #	Distance from umbo (mm)	Conventional ^{14}C age (BP) ±1σ	δ^{13}C (‰, PDB)	Marine model age (BP) ±1σ	ΔR ±1σ
Beta-284384	~1–11	600 ± 40	–2.50	456 ± 23	144 ± 46
OS-95242	41	500 ± 25	–2.14	456 ± 23	44 ± 34
OS-95243	44	540 ± 25	–1.93	456 ± 23	84 ± 34
				Average ΔR	81 ± 29
				Range	100

The archaeological samples provided a similar range of variability as the historical samples (Table 2, Figure 2). The lowest amount of variability came from the three samples from 44NH478. These samples were taken 52, 56, and 65 mm from the shell umbo. The conventional ages vary by 60 ^{14}C yr and the average of all the samples is 3103 ± 17 ^{14}C yr ($T = 2.12$, $\chi^2_{0.05} = 5.99$), suggesting reasonable agreement between the samples. The 1σ calibrated age ranges also have significant overlap.

Table 2 ^{14}C data from archaeological *Crassostrea virginica* shells analyzed in this study.

Sample #	Lab # (OS-)	Provenience	Distance (mm from umbo)	δ^{13}C (‰, PDB)	^{14}C age (BP) ±1σ	cal BP (1σ)	cal BP (2σ)
18DO130, Fishing Bay, Maryland, USA							
DO130-1	94972	Pit feature, 10 cm	72[a]	–1.44	1860 ± 35	1550–1430	1610–1380
DO130-4	95093	Pit feature, 10 cm	72[a]	–0.9	1770 ± 25	1450–1340	1500–1310
DO130-3	95092	Pit feature, 10 cm	74	–1.18	1800 ± 25	1490–1390	1520–1340
DO130-2	95091	Pit feature, 10 cm	75	–1.11	1860 ± 25	1550–1440	1600–1390
				Range	90		
18DO429, Fishing Bay, Maryland, USA							
DO429-1	95094	Unit 1, Stratum 1	75	–3.68	1270 ± 25	950–870	980–790
DO429-4	95097	Unit 1, Stratum 1	78	–3.29	1340 ± 25	1010–920	1060–900
DO429-3	95096	Unit 1, Stratum 1	83	–2.88	1320 ± 25	990–910	1050–890
DO429-2	95095	Unit 1, Stratum 1	90	–1.96	1370 ± 30	1050–950	1110–920
DO429-5	101201	Unit 1, Stratum 1	110	–2.82	1350 ± 25	1030–940	1070–910
				Range	100		
44NH478, near the Chesapeake Bay mouth, Virginia, USA							
NH478-1	95244	Unit 1, bulk sample, 20 cm	52	0.34	3070 ± 40	2910–2760	3000–2720
NH478-2	95249	Unit 1, bulk sample, 20 cm	56	–0.41	3090 ± 25	2920–2780	3000–2740
NH478-3	101203	Unit 1, bulk sample, 20 cm	65	–0.66	3130 ± 25	2990–2830	3060–2770
				Range	60		

[a] OS-94972 and OS-95093 were both taken at a distance of 72 mm from the umbo, but were taken about 2 mm apart perpendicular to the umbo.

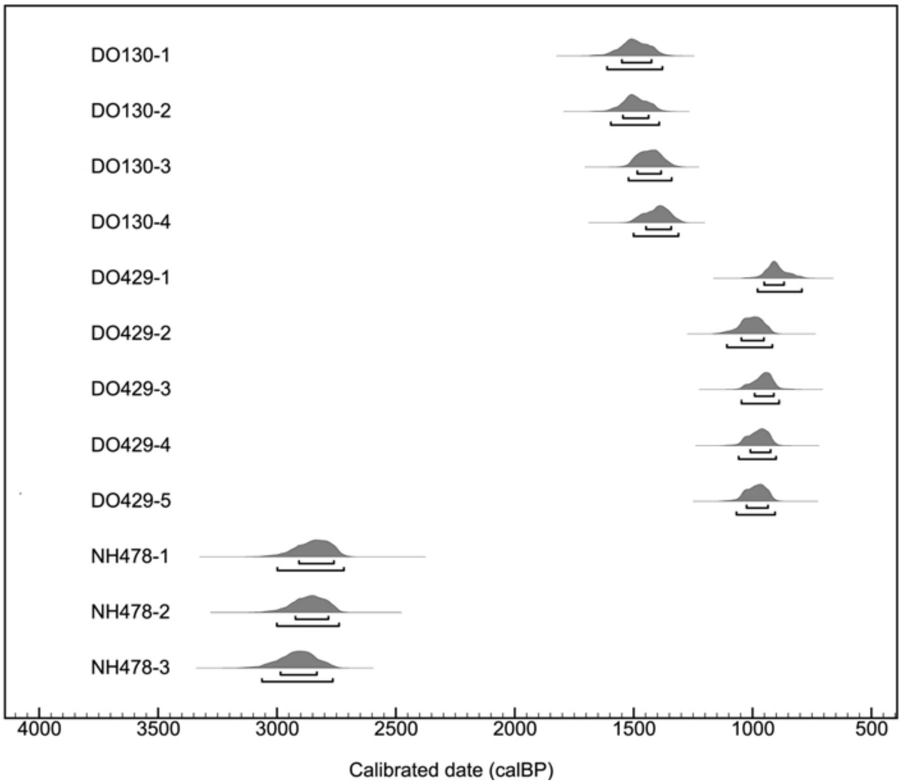

Figure 2 Calibrated ^{14}C ages (1σ and 2σ noted in the bars above) for 12 intrashell archaeological samples reported in Table 2. Calibrated with OxCal v 4.2 (Bronk Ramsey 2009) using the Marine09 calibration curve (Reimer et al. 2009). For all DO samples, a ΔR of –88 ± 23 yr was applied; for NH samples, a ΔR of 2 ± 46 yr was applied (Rick et al. 2012).

Four samples from the *C. virginica* valve from 18DO130 provided a range of about 90 ^{14}C yr in the conventional ^{14}C date. These samples were taken from between 72 to 75 mm from the umbo. The samples were obtained at relatively similar distances because, like other specimens from the site, the shell had areas of apparent dissolution from boring sponges, which may have resulted in localized recrystallization. Although at a similar distance from the umbo, these specimens were obtained ~2 mm apart perpendicular to the shell growth axis, providing a slight lateral range of ^{14}C variability. The average of all 3 samples is 1817 ± 23 ^{14}C yr ($T = 8.46$, $\chi^2_{0.05} = 7.81$), with χ^2 values suggesting a slightly wider dispersion than expected. When these ^{14}C dates are corrected and calibrated, all overlap at 1σ.

At 18DO429, five samples provided a range of 100 ^{14}C yr. These were obtained from distances between 75 and 110 mm from the umbo. The average of all five samples is 1327 ± 17 ^{14}C yr ($T = 8.45$, $\chi^2_{0.05} = 9.49$) with reasonable agreement between the samples. Like the other samples, the 1σ calibrated age ranges all overlap.

CONCLUSIONS

Previous research has documented significant intrashell variability in ^{14}C content in mollusks from California and Peru (Culleton et al. 2006; Jones et al. 2007, 2010) and more limited variability in specimens from Georgia's Atlantic coast (Thomas et al. 2013). The variability in ^{14}C from Peru and California is thought to stem largely from seasonal variations in coastal upwelling, but both Culleton et al. (2006) and Jones et al. (2007) demonstrate through shell $\delta^{18}O$ and $\delta^{13}C$ compositions that seasonal fluctuations do not always correlate with high variability in ^{14}C ages. While calibration tends to reduce the biases of intrashell variability, these discrepancies can pose challenges for researchers seeking to build high-precision chronologies (Culleton et al. 2006).

Our ^{14}C dates from the Chesapeake Bay *C. virginica* shells provide similar results to those reported from the Georgia coast (~80 ^{14}C yr or less; Thomas et al. 2013). The historical sample from Point Lookout, Maryland, documented variability in ΔR as high as 100 yr, and archaeological samples documented a range of 60–100 ^{14}C yr, much lower than the Pacific coast values (Culleton et al. 2006; Jones et al. 2007). Despite this more limited variability, researchers should still use caution when subsampling Chesapeake Bay mollusk shells for AMS ^{14}C analysis. Differences between ^{14}C dates in individual shells are still present and potentially important for understanding broader carbon cycling in the Chesapeake Bay. The discrepancies observed in these 15 samples may result from ^{14}C-depleted freshwater that enters the bay from some if its drainages, ^{14}C-depleted seawater that enters the bay at its mouth, and/or biological carbon recycling (Rick et al. 2012). Future intrashell dating of a larger number of samples, complemented by stable isotope analysis from the same specimens, may prove fruitful for better understanding these processes.

This study's focus was on how intrashell variability in ^{14}C content may affect archaeological and geological ^{14}C chronology building and interpretation, with the results herein demonstrating that intrashell variability is a potential source of bias for building high-precision AMS ^{14}C chronologies using small subsamples of mollusk shells. These effects seem to be relatively limited along the North American Atlantic coast for *C. virginica* and *M. mercenaria* shells, especially for ^{14}C reservoir age-corrected and calibrated ^{14}C age estimates. Culleton et al. (2006) advocate taking relatively large subsamples of shells from ~1–2 cm perpendicular to the growth axis and preferably from the distal ends of larger, presumably older specimens. We also advocate this straightforward sampling procedure as a precautionary measure to help minimize the potential of intrashell ^{14}C variability. Because intrashell variability is now known to differ geographically, and because the scope of these studies has been relatively small, we recommend researchers continue to test for potential intrashell biases for ^{14}C dating in other areas around the world.

ACKNOWLEDGMENTS

Funds for ^{14}C dating were provided by the National Museum of Natural History Small Grants Program. Our fieldwork at 18DO130 and 18DO439 was supported by a Smithsonian Institution Restricted Endowment Grant and our work at 44NH478 was supported by the Virginia Department of Historic Resources Threatened Sites Program. Ellen Strong and Paul Greenhall of the Department of Invertebrate Zoology, National Museum of Natural History, Smithsonian Institution, graciously provided access to the historical specimen from Point Lookout. We thank Mike Barber, Charlie Hall, Dave Hazzard, Darrin Lowery, and John Wah for their support and collaboration. Finally, we acknowledge A J T Jull, Mark McClure, the editorial staff of *Radiocarbon*, Doug Kennett, and anonymous reviewers for comments on this manuscript.

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