CENTURIES OF MARINE RADIOCARBON RESERVOIR AGE VARIATION WITHIN ARCHAEOLOGICAL *MESODESMA DONACIUM* SHELLS FROM SOUTHERN PERU

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ABSTRACT. Mollusk shells provide brief (<5 yr per shell) records of past marine conditions, including marine radiocarbon reservoir age (*R*) and upwelling. We report 21 ^{14}C ages and *R* calculations on small (~2 mg) samples from 2 *Mesodesma donacium* (surf clam) shells. These shells were excavated from a semi-subterranean house floor stratum ^{14}C dated to 7625 ± 35 BP at site QJ-280, Quebrada Jaguay, southern Peru. The ranges in marine ^{14}C ages (and thus *R*) from the 2 shells are 530 and 170 ^{14}C yr; *R* from individual aragonite samples spans 130 ± 60 to 730 ± 170 ^{14}C yr. This intrashell ^{14}C variability suggests that ^{14}C dating of small (time-slice much less than 1 yr) marine samples from a variable-*R* (i.e. variable-upwelling) environment may introduce centuries of chronometric uncertainty.

INTRODUCTION

The marine radiocarbon reservoir age, *R*, is the ^{14}C age of the carbon dissolved in seawater (Stuiver et al. 1986). Many marine mollusks precipitate their calcitic or aragonitic shells from this carbon, with only a minor contribution from metabolized food carbon (Lorrain et al. 2004; Gillikin et al. 2007). *R* can be calculated from the ^{14}C age of a mollusk shell if the time since its death is known, e.g. through historical records or independent chronometry, as

$$R(t) = M_m(t) - A(t)$$

where $M_m(t)$ is the measured ^{14}C age of a marine mollusk of known calendar age t, and $A(t)$ is the atmospheric ^{14}C age, obtained from a terrestrial calibration curve such as IntCal04 (Reimer et al. 2004). Conversely, ^{14}C chronometry on marine mollusk shell is possible when local *R* is known. Marine ^{14}C chronometry can be difficult, because *R* is often not known with certainty: it changes geographically and temporally, and these changes are poorly constrained.

Marine upwelling has a strong effect on local *R* in areas such as the Peruvian coast, where upwelling is variable (Huyer et al. 1987; Jones et al. 2009). The few existing estimates of marine reservoir age in southern Peru during the early Holocene vary by nearly 1000 ^{14}C yr (Owen 2002; Fontugne et al. 2004). Additionally, investigations of *R* at high temporal resolution, by examining ^{14}C changes within individual shells (Andrus et al. 2005; Culleton et al. 2006; Jones et al. 2007, 2009), show over 200 ^{14}C yr of seasonal *R* variation (i.e. within a single year of growth) in modern shells from coastal Peru.

To better understand *R* and its variability in southern Peru, we investigated early Holocene *R* and its intra-annual changes there. We report ^{14}C dates on charcoal and on 21 samples from 2 associated *Mesodesma donacium* (surf clam) shells, excavated from site Quebrada Jaguay 280 (QJ-280), Peru (Sandweiss et al. 1998), and *R* calculations based on these ^{14}C dates. We discuss the observed intrashell *R* variation, compare our *R* values with those of others, and discuss possible reasons for and chronometric implications of *R* variability.

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STUDY SITE AND SAMPLES

Site QJ-280 is located at 16°30′S, near Camaná in southern Peru (Figure 1). The site is on an alluvial terrace just west of Quebrada Jaguay itself and 2 km from the coast (Sandweiss et al. 1998; Tanner 2001). Occupation extends from the Terminal Pleistocene (e.g. BGS-1942, 11,110 ± 260 BP) to the early Holocene (e.g. BGS-1700, 7500 ± 130 BP; both dates from Sandweiss et al. 1998), and can be divided into distinct Terminal Pleistocene and early Holocene components.

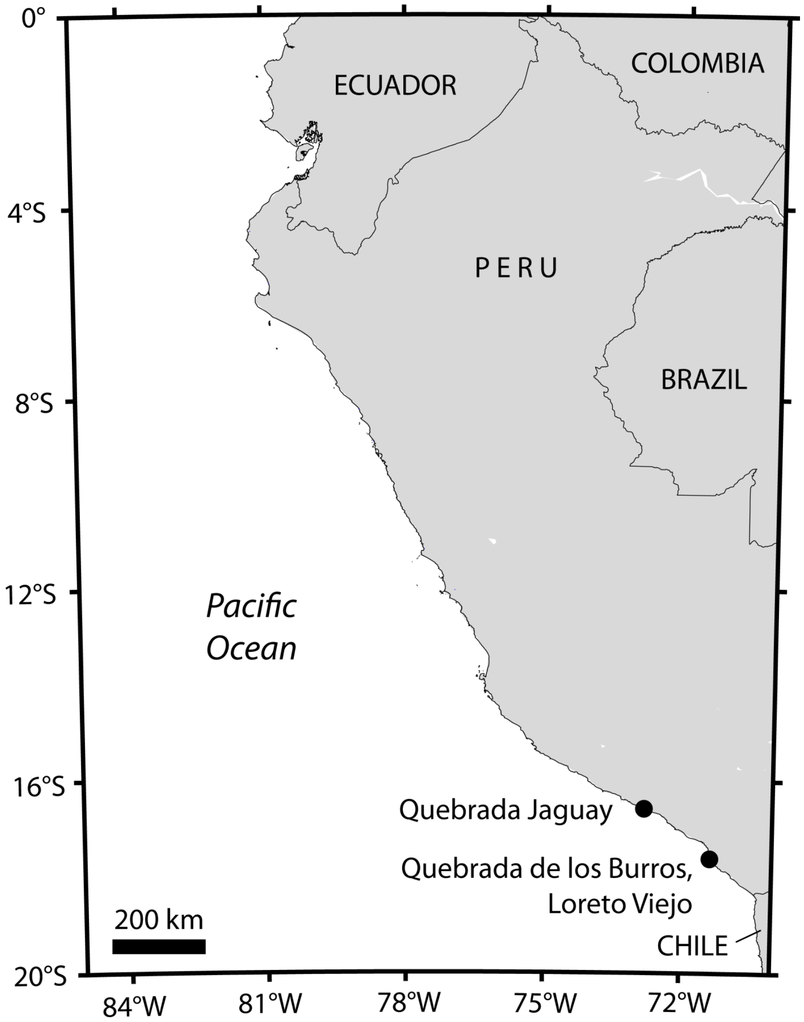

Figure 1 Map showing locations of sites discussed in the text

We investigated a floor stratum of a semi-subterranean house from the early Holocene component of Sector I of the site. The house deposit consists of 6 floor layers (1a2–1f from top to bottom) containing charcoal and plant material. Five ^{14}C dates on charcoal and gourd fragments from layers 1b–1e all overlap at 1 σ, suggesting that the layers were deposited in rapid succession (Sandweiss et al. 1998; Tanner 2001; Erickson et al. 2005). The basal house-floor stratum (1f) dates slightly older than the overlying floors; excavation reports suggest that charcoal from this layer is probably a mixture from the floor and from older underlying deposits. We investigated the previously undated floor layer 1a2.

In addition to charcoal, layer 1a2 contained 2 complete *M. donacium* valves from different individuals. This species of surf clam lives <10 m below sea level along the Peruvian and Chilean coasts (Tarifeño-Silva 1980) and has been harvested in the surf zone for food by humans for thousands of years (Sandweiss et al. 1998). Clams precipitate shell aragonite in equilibrium with seawater (Mook and Vogel 1968), forming sequential growth bands that record environmental conditions and periodicity (Clark 1974), including marine ^{14}C content (Taylor and Berger 1967).

Field archaeologists supplied 2 *M. donacium* shells and terrestrial-plant-derived charcoal from QJ-280, unit I-3-B, level 1a2. These samples were interpreted provisionally by the archaeologists as contemporaneous, and thus suitable for *R* calculations, based on site stratigraphy.

METHODS

We measured the ^{14}C ages of 2 separate pieces of charcoal from layer 1a2 as a check on the contemporaneity of material in the stratum. Dissimilar ages would suggest that deposition of the layer was insufficiently rapid for meaningful *R* calculations. The samples received standard acid-base-acid (ABA) pretreatment to remove any carbonates, fulvic acids, and humic acids. The samples were dried, combined with copper oxide, placed in a sealed and evacuated tube, and then combusted by heating in isolation. The CO_2 produced was cryogenically isolated and graphitized using standard procedures (Slota et al. 1987). Dating, including isotope-ratio and sample-size corrections (Donahue et al. 1990), was carried out at the NSF-Arizona AMS Facility, Tucson, Arizona, USA.

We dated aragonite powder samples (14 from shell 1, 7 from shell 2), ~2 mg each, from the *M. donacium* valves. Shell aragonite was powdered along troughs following growth lines (to minimize the amount of time averaged within individual samples) to a depth less than 0.5 mm using a carbide dental bur 0.5 mm in diameter, after briefly abrading the shell surface to remove any surficial contamination. We reacted the aragonite with phosphoric acid *in vacuo* at room temperature. The reaction was allowed to proceed to completion and the CO_2 produced was cryogenically isolated and ^{14}C dated as above.

We also sampled the shells for $\delta^{18}O$ and $\delta^{13}C$ at multiple locations (165 from shell 1, 38 from shell 2) along axes from umbo to edge. Aragonite from shell 1 was powdered using an 0.08-mm-diameter carbide bur and Merchantek/New Wave Research micromill. The outer 10 µm of shell was milled and discarded to remove possible contamination; samples were then milled to an additional depth of 150 µm, reacted with dehydrated phosphoric acid under continuous helium flow, and analyzed using a Thermo Scientific GasBench II coupled to a Delta Plus isotope-ratio mass spectrometer at the University of Alabama. At least one NBS-19 standard was run for every 6 shell aragonite samples. We powdered aragonite from shell 2 in divots <0.5 mm deep with a 0.5-mm-diameter carbide bur, then reacted the samples with dehydrated phosphoric acid *in vacuo* and analyzed them for $\delta^{18}O$ and $\delta^{13}C$ using a Finnigan MAT 252 mass spectrometer with a Kiel III automated sampling device at the University of Arizona. At least 1 internal standard of NBS-19 calcite was run for every 19 shell aragonite samples. One-sigma precision on repeated standards is ±0.1‰ for $\delta^{18}O$ and ±0.06‰ for $\delta^{13}C$.

RESULTS

The charcoal samples dated to 7600 ± 40 and 7670 ± 60 BP (Table 1), overlapping at 1 σ. ^{14}C ages for 14 carbonate samples from *M. donacium* shell 1 are between 7820 ± 100 and 8350 ± 170 BP (Figure 2a), and for 7 samples from shell 2 are between 7750 ± 50 and 7920 ± 50 BP (Table 3,

Figure 2b). Based on a weighted average terrestrial age of 7625 ± 35 BP for the level containing the clams, *R* estimates over the life of clam 1 are 200 ± 110 to 730 ± 170 ^{14}C yr, a range of 530 ± 200 ^{14}C yr (Table 2). The weighted mean *R* is 360 ^{14}C yr. Similarly, *R* estimates for clam 2 are 130 ± 60 to 300 ± 80 ^{14}C yr, a range of 170 ± 100 ^{14}C yr. The weighted mean *R* is 230 ^{14}C yr (Table 3).

Table 1 Terrestrial ^{14}C ages from stratum I-3-B level 1a2, site QJ-280. Calibration calculations were performed using OxCal v 4.0.5 (Bronk Ramsey 1995, 2001) and the SHCal04 calibration curve (McCormac et al. 2004). Where more than 1 calibrated age range is reported for a sample, probability (*p*) is given for each range.

Lab nr	Material	δ^{13}C (‰)	^{14}C age ±1 σ (BP)	2-σ calibrated age (cal BP)
AA-75279	plant charcoal	–24.7	7600 ± 40	8420–8290 $p = 0.85$ 8260–8210 $p = 0.10$
AA-75280	plant charcoal	–24.4	7670 ± 60	8540–8330

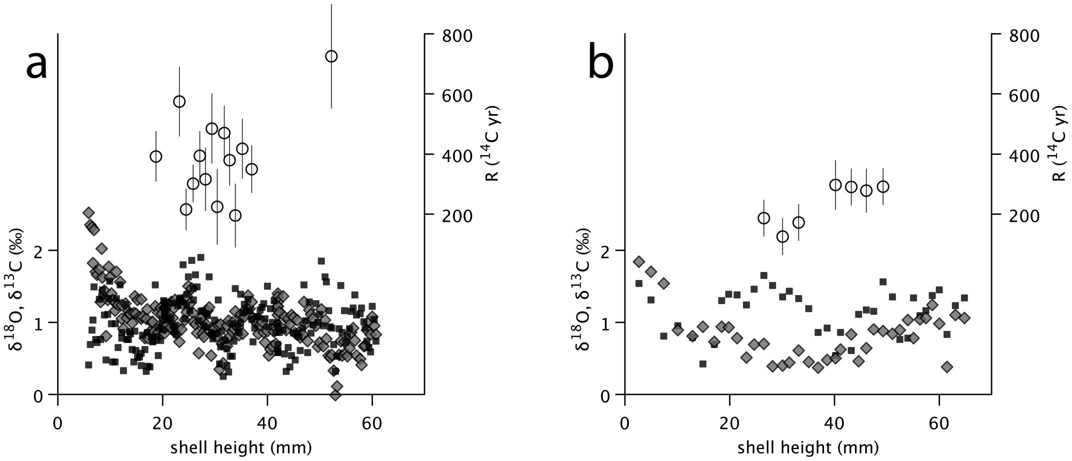

Figure 2 Marine ^{14}C reservoir age (*R*, open circles), δ^{18}O (black squares), and δ^{13}C (gray diamonds) profiles from *M. donacium* shells excavated from stratum 1a2, site QJ-280, Quebrada Jaguay, Peru. Shell 1 is shown in (a); shell 2 is shown in (b). Error bars on *R* are 1 σ; 1-σ errors on δ^{18}O and δ^{13}C measurements are approximately the size of the diamonds.

The δ^{18}O of shell 1 ranges from 0.3 to 1.9‰ (VPDB). Four to 5 cycles in δ^{18}O are apparent, although they are difficult to distinguish clearly near the distal edge of the shell (Figure 2a). The δ^{18}O of shell 2 ranges from 0.4 to 1.7‰ (VPDB). Shell 2 exhibits 3 to 4 cycles in δ^{18}O; these are also compressed and unclear near its distal edge (Figure 2b). The δ^{13}C of shell 1 ranges from 0.0 to 2.6‰ (VPDB) and follows cycles similar to but less clear than those of δ^{18}O, superimposed on a trend of decreasing δ^{13}C with shell height. The δ^{13}C of shell 2 ranges from 0.3 to 1.9‰ (VPDB), and is greatest near the umbo.

DISCUSSION

Intrashell ^{14}C age range (i.e. intrashell *R* range) may be a useful proxy for variability in marine upwelling, as greater *R* is due to greater upwelling of deep, ^{14}C-depleted water in an environment where upwelling varies dynamically. The 530 ^{14}C yr age range within shell 1 is larger than any previously reported in a mollusk shell, suggesting large changes in southern Peruvian upwelling during

Table 2 ^{14}C ages of aragonite and calculated marine reservoir ages (R) from *M. donacium* shell 1, excavated from I-3-B level 1a2, site QJ-280.

Lab nr	Shell height (mm)	^{14}C age ±1 σ (BP)	R ±1 σ
AA-79558	18.7	8020 ± 80	390 ± 80
AA-79559	23.2	8200 ± 110	580 ± 120
AA-81665	24.5	7840 ± 60	220 ± 70
AA-81666	25.8	7930 ± 50	300 ± 60
AA-79560	27.1	8020 ± 70	390 ± 80
AA-80391	28.2	7940 ± 100	320 ± 110
AA-80392	29.4	8110 ± 110	490 ± 120
AA-79561	30.4	7850 ± 120	230 ± 130
AA-80393	31.8	8100 ± 80	470 ± 90
AA-80394	32.8	8010 ± 80	380 ± 80
AA-79562	33.9	7820 ± 100	200 ± 110
AA-80395	35.2	8040 ± 90	420 ± 100
AA-80396	37.0	7980 ± 70	350 ± 80
AA-79563	52.2	8350 ± 170	730 ± 170

Table 3 ^{14}C ages of aragonite and calculated marine reservoir ages (R) from *M. donacium* shell 2, excavated from I-3-B level 1a2, site QJ-280.

Lab nr	Shell height (mm)	^{14}C age ±1 σ (BP)	R ±1 σ
AA-86411	26.5	7810 ± 50	190 ± 60
AA-86412	30.1	7750 ± 50	130 ± 60
AA-86413	33.2	7800 ± 50	170 ± 60
AA-86414	40.2	7920 ± 70	300 ± 80
AA-86415	43.2	7920 ± 50	290 ± 60
AA-86416	46.1	7900 ± 60	280 ± 70
AA-86417	49.3	7920 ± 50	290 ± 60

the brief (<5 yr) life of this clam. The ^{14}C age range within shell 2 (170 ^{14}C yr) is less, and closer to the intrashell variation in early 20th-century Peruvian *A. purpuratus* shells (120 ± 70 to 290 ± 80 ^{14}C yr of variation within individual shells) reported by Jones et al. (2009). Observed intrashell variation can be considered a minimum for the total R variation a mollusk experienced through its life; additional, unobserved variability may exist in unsampled portions of shell.

R within a shell typically correlates with shell δ^{18}O (in part a water temperature proxy, Grossman and Ku 1986) in Peruvian mollusks. This relationship is clearest in shells from the late 20th century, when "bomb carbon" increased the R contrast between surficial and deep marine water (Andrus et al. 2005), and is detectable with more difficulty in early 20th-century shells (Jones et al. 2009). The early Holocene shells discussed here, however, show no clear relationship between δ^{18}O and R. Some scatter in ^{14}C data from archaeological shells may result from diagenetic alteration, although visual microscopy and X-ray powder diffraction analyses show no evidence of alteration in our shells, and δ^{18}O profiles do not show the loss of definition that diagenesis could produce. It is possible that our ^{14}C samples, although <0.5 mm deep, may have extended through the outer layer (Carré et al. 2005) and incorporated some reworked inner shell material of slightly different ^{14}C age, obscuring the trend of interest. Decay of shell ^{14}C over time may have obscured some of the R-δ^{18}O relationship by decreasing the ^{14}C sample-to-background ratio and thus the precision of ^{14}C determinations.

Absolute *R* estimates using modern marine mollusk shells with known collection dates can be as accurate as the shell ^{14}C data. Absolute *R* estimates from prehistoric shells are only as accurate as the estimate of shell antiquity, difficult to quantify with certainty, even in undisturbed and well-understood archaeological contexts. Differences in *R* estimates between shells may result from differences in marine conditions or simply from errors in ascertaining the dates of mollusk death and subsequent shell deposition. Comparisons of *R* estimates from QJ-280 with those from other studies in southern Peru and northern Chile illustrate some of this effect.

Early Holocene *R* estimates calculated from the QJ-280 shells are similar to or slightly younger than those from multiple small samples of early 20th-century bay scallop (*Argopecten purpuratus*) shells (360–615 ^{14}C yr; Jones et al. 2007, 2009). Our *R* results from QJ-280 are also similar to or slightly younger than those (450–540 ^{14}C yr) of Southon et al. (1995) for northern Chile (20ºS) in the late Holocene (1.8–1.1 cal kyr BP), calculated using paired pelican feathers and skin and camelid-hair yarn from a feathered garment, and paired sea lion or seal tissue and terrestrial-plant-fiber thread from a water bottle. *R* estimates for the QJ-280 *M. donacium* shells are generally younger than those reported by Owen (2002) from Loreto Viejo, southern Peru (17ºS). Owen reports reservoir ages 630–870 ^{14}C yr at 0.7 cal kyr BP and 520 ^{14}C yr at 3.7 cal kyr BP, using paired whole *C. chorus* shells and small twigs. Two reservoir ages over 2000 ^{14}C yr were dismissed as erroneous. Based on 13 pairs of ^{14}C ages on charcoal and whole shells from Quebrada de los Burros (Figure 1) and nearby sites, Fontugne et al. (2004) report highly variable marine reservoir ages of 480–1290 ^{14}C yr through the Holocene. The weighted mean *R* estimates from the QJ-280 shells are lower than all *R* estimates from Fontugne et al. (2004), in one case by more than 1000 ^{14}C yr.

Why are the *R* estimates from the two QJ-280 clam shells lower than other local *R* estimates? Perhaps these clams lived during times or in locations of anomalously little upwelling, as upwelling patterns may change from year to year. Bubbles and turbulence from waves breaking in the surf zone greatly enhance air-sea carbon exchange (Wallace and Wirick 1992; Farmer et al. 1993), which may cause nearshore *R* to approach the atmospheric ^{14}C age more closely than *R* in the open ocean. Perhaps surf zone bubbles caused anomalously young ^{14}C ages in these surf clams, and are partially responsible for the low *R* in the QJ-280 clams.

Summer river discharge from high-altitude precipitation or snowmelt, with different ^{14}C content than that of the open ocean, could also produce anomalous *R* (either young from modern precipitation, or old from melting of ancient glacial ice) near river mouths. Site QJ-280 is located 20 km northwest of the perennial Río Camaná and immediately west of the typically dry Quebrada Jaguay. The δ^{18}O of this river water would be very low (<–12‰, Thompson et al. 1986) relative to that of the ocean (~0‰), however, and an influx of river water large enough to alter *R* would also substantially deplete the δ^{18}O of mollusk shell carbonate. We see no evidence of such δ^{18}O in the QJ-280 clams, and so do not believe that a summer influx of river water could account for their low *R* values.

Finally, the discrepancies between our data and those of Fontugne et al. (2004) may also illustrate difficulties in identifying truly contemporaneous sample pairs. Mollusk death and deposition centuries after deposition of the charcoal in stratum 1a2 could also explain the low *R* estimates from the two QJ-280 clams. The thickness and stratigraphic integrity, confirmed by ^{14}C dating (Sandweiss et al. 1998; Tanner 2001), of the 1a2–1e stratigraphic package makes this unlikely, however. Fontugne et al. (2004) recognized the importance of contemporaneity of marine and terrestrial samples for reservoir age calculations, and selected very small (<1 mm) grains of "microcharcoal" from inside mollusk shells to ensure that the components of their sample pairs were contemporaneous. It is possible that charcoal grains this small may have diffused downward from younger overlying strata over time. Using such anomalously young charcoal powder in reservoir age calculations would produce anomalously old and possibly variable marine reservoir ages, such as those in the Fontugne et al. (2004) data.

Although the presence of ^{14}C centuries of intrashell variation warrants caution when using marine shells—particularly small bits of shell—for ^{14}C chronometry in Peru or another area with seasonally variable marine upwelling, intrashell ^{14}C variation may be a viable short-term, high-frequency proxy for marine upwelling variability in an environment like coastal Peru. The ^{14}C centuries of intrashell ^{14}C variability reported here suggest that southern Peru experienced dynamic changes in upwelling on seasonal or shorter timescales during the early Holocene.

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