

## INFLUENCE OF MOLLUSK SPECIES ON MARINE $\Delta R$ DETERMINATIONS

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**ABSTRACT.** Radiocarbon ages were measured on replicate samples of burnt grain and 5 mollusk species collected from a single sealed layer at an archaeological site (Hornish Point) on the west coast of South Uist, Scotland. The aim was to examine the impact of using different mollusk species on  $\Delta R$  determinations that are calculated using the paired terrestrial/marine sample approach. The mollusk species examined inhabit a range of environments and utilize a variety of food sources within the intertidal zone. Several authors have suggested that these factors may be responsible for observed variations in the  $^{14}\text{C}$  activity of mollusk shells that were contemporaneous in a single location. This study found no significant variation in the  $^{14}\text{C}$  ages of the mollusk species, and consequently, no significant variation in calculated values of  $\Delta R$ . The implication is that in an area where there are no carboniferous rocks or significant local inputs of freshwater to the surface ocean, any of a range of marine mollusk species can be used in combination with short-lived terrestrial material from the same secure archaeological context to accurately determine a  $\Delta R$  value for a particular geographic location and period in time.

### INTRODUCTION

Marine mollusk shell carbonate is an effective record of changes in certain isotopic ratios in the ambient water; therefore, it is extensively used to provide data for paleoenvironmental reconstructions. For example, the  $^{18}\text{O}/^{16}\text{O}$  ratio is determined by water temperature at the time of precipitation (Epstein et al. 1953; Grossman and Ku 1986) and can be used as a proxy to examine past variability in sea surface temperature. Radiocarbon age measurements made on mollusk shells are often used to give a chronological context to these records, and this also enables comparisons to be made between different data sets. In addition to being a resource for paleoenvironmental data, marine mollusk shells are abundant in many regions of coastal archaeological remains, and  $^{14}\text{C}$  age measurements may be made on them when there is an absence of secure, datable terrestrial material. A major consideration when using these  $^{14}\text{C}$  measurements to establish a chronological framework for an archaeological site is the  $^{14}\text{C}$  depletion of the oceans relative to the terrestrial biosphere—the so-called marine reservoir effect (MRE).

The MRE can be modified locally by such factors as the influx of terrestrial carbon sources including organic carbon from terrestrial run-off, dissolved geological carbonates, and, in the case of major rivers, a relatively modern inorganic carbon input via  $\text{CO}_2$  exchange into river water. The effect is amplified in areas of enclosed coastal topography (e.g. fjords) where circulation with the open ocean is limited and is reflected in shell carbonates precipitated in affected areas (Heier-Neilsen et al. 1995). Mollusk shell  $^{14}\text{C}$  activity can also be modified by exchange with environmental carbon (e.g. carbonates within percolating groundwater; Bezerra et al. 2000) during postmortem recrystallization. Thus, rigorous inspection and pretreatment is required to avoid contaminated samples.

It is well established that the oceans are depleted in  $^{14}\text{C}$  relative to the atmosphere and that this depletion varies both spatially and temporally. This depletion is translated to organisms that incorporate marine carbon, including mollusks. The carbonate shell of these organisms contains carbon derived from the dissolved inorganic carbon component (DIC) of the surrounding water mass, and its specific  $^{14}\text{C}$  activity will reflect the MRE. The depletion exists because of the constant removal of surface water to the deep ocean by density-driven downward circulation, particularly in the polar

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regions. This removes a water mass from the atmosphere-ocean interface and therefore prevents further gaseous exchange of CO<sub>2</sub> with the atmosphere. Such a water mass may then be resident in the deep oceans for an extended period, during which time its <sup>14</sup>C activity decreases due to radioactive decay. Upwelling eventually returns deep water to the surface ocean, which in turn depletes the surface water <sup>14</sup>C activity. The consequence of a MRE is that <sup>14</sup>C measurements made on samples that contain marine-derived carbon must be corrected to be comparable with values for coeval atmospheric carbon. Currently, marine sample ages can be calibrated with the MARINE04 calibration data set (Hughen et al. 2004), which provides an average time-dependent correction for the age of the global surface ocean. The deviation of marine <sup>14</sup>C ages at a specific location (and time) from this global average is known as  $\Delta R$  and varies geographically as a function of local climatic and oceanic variables (Stuiver and Braziunas 1993; Stuiver et al. 1998).  $\Delta R$  can be calculated by empirical measurement of local samples, and a variety of approaches have been adopted (Ascough et al., forthcoming). One method is the comparison of contemporaneous marine and terrestrial material obtained from terrestrial deposits such as coastal midden sites. This is known as the paired sample method.

Measurements of marine mollusk shells are often used to determine  $\Delta R$ . In the paired sample approach, this material has the advantage of being produced by generally short-lived, relatively sessile organisms, and also of being abundant in both marine and coastal deposits. As a result, the shells of many different species have been used to determine  $\Delta R$  at various locations. However, several authors have suggested that species-dependent variations in shell <sup>14</sup>C activity mean that significantly different MREs (and therefore  $\Delta R$  values) can be derived from measurements made on various mollusk species at a single location (Forman and Polyak 1997; Hogg et al. 1998). In contrast, Harkness (1983) found no clear species-dependent variations in natural enrichment in an assessment of modern UK coastal MRE. Different marine mollusk species inhabit a range of ecological niches within the intertidal zone and deeper ocean where a variety of feeding mechanisms and food sources are utilized. These differences are commonly identified as responsible for observed interspecies variation in shell <sup>14</sup>C activity. For example, Ingram (1996) suggests that species-dependent variations in mollusk shell <sup>14</sup>C content may be a function of differing food sources and seasonal growth patterns allied with circulation and upwelling. If a specific feeding mechanism or habitat means that carbon incorporated into shell CaCO<sub>3</sub> has a different <sup>14</sup>C activity to that of other species, this would result in different calculated  $\Delta R$  values using the paired terrestrial/marine sample approach.

The majority of shell carbonate is precipitated from dissolved inorganic carbon (DIC) in the water column, with a variable portion derived from metabolic sources (Tanaka et al. 1986; Dettman et al. 1999). Proportionally higher amounts of metabolically derived carbon appear to be contained within the soft tissues (Uerpmann 1990). While the DIC at a specific location is relatively homogeneous, metabolic carbon resources differ between species depending upon habitat and feeding mechanism. Mollusk habitats include the hard substrate (e.g. bedrock outcrops), the sediment surface (epifaunal position), and below the sediment surface (infaunal position), while feeding mechanisms include grazing upon microalgae, detritus, and seaweeds, or filter-feeding on organic material suspended in the water column. In the absence of significant local terrestrial inputs, the suspended material (plankton, etc.) that is utilized by filter feeders usually has a <sup>14</sup>C activity that is closer to ocean DIC and may mean that filter feeders (e.g. mussels and oysters) incorporate proportionally lower amounts of atmospheric <sup>14</sup>C than herbivorous grazing species (e.g. limpets and periwinkles). The latter consume seaweeds that contain carbon derived from the atmosphere when photosynthesis proceeds while the seaweed is exposed at low tide. It is also possible in areas where there is a significant source of geological carbon for this to be incorporated into shell structure during growth, as sedimentary particles are taken up by the mollusk during grazing (Dye 1994), or while inhabiting car-

bonate-rich sediments (Forman and Polyak 1997). Dyke et al. (2002) suggest that the elevated  $^{14}\text{C}$  age of the deposit-feeding marine mollusk *Portlandia arctica* is the result of its infaunal position and feeding mechanism, while Forman and Polyak (1997) observed that mollusks with sessile habitats and pelagic food sources gave significantly lower MRE offsets (i.e. a younger  $^{14}\text{C}$  age).

This paper examines the variation in  $^{14}\text{C}$  age and values of  $\Delta R$  that can be derived from mollusk shells of 5 different species from a single archaeological deposit. The deposit is located in an Iron Age site at Hornish Point on the west coast of the island of South Uist, Scotland (Figure 1) in an area where there is no significant local input of terrestrial-source carbonate to the surface ocean water.

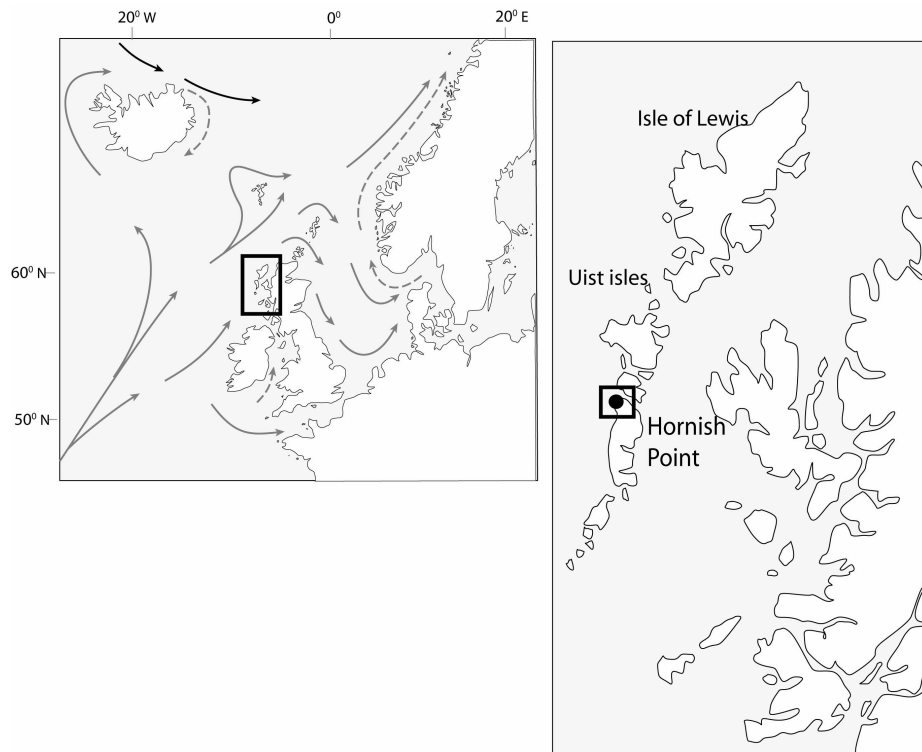


Figure 1 Location of Hornish Point within the North Atlantic. Warm Atlantic currents are shown in gray, cold currents in black, and coastal waters as gray dashed lines.

## METHODS

Samples of carbonized cereal grain and marine mollusk shell were taken from a single sealed layer within a midden deposit on a headland exposed to the open ocean and away from significant sources of freshwater or carbonate geology. The midden had accumulated rapidly and contained a particularly high concentration of carbonized grain and marine mollusk shells of several species, making it suitable for the paired sample approach to determining  $\Delta R$ .

Four individual carbonized barley (*Hordeum* sp.) grains were taken for  $^{14}\text{C}$  measurement, together with 5 different species of mollusk shells. These were common limpet (*Patella vulgata*), common mussel (*Mytilus edulis*), common cockle (*Cerastoderma edule*), razor shell (*Ensis ensis*), and common periwinkle (*Littorina littorea*). Four shells were taken of each species except for *Cerastoderma*

*edule* where 3 shells were taken. This was due to the lower density of intact whole shells of this species in the context, and although shell fragments were available, these were avoided to exclude the possibility of inadvertently measuring the same shell twice. For the same reason, only the left-hand shell portions of bivalve species were selected for analysis.

Standard pretreatment methods were used for both the grain and shell samples. For the grain, this involved acid-alkali-acid extraction of contaminants followed by sample combustion in evacuated sealed quartz tubes (Vandeputte et al. 1996) using copper oxide as the oxidant. Before pretreatment, the shells were inspected and only non-porous specimens with preserved textures were selected for analysis (Mangerud 1972; Mook and Waterbolk 1985). Pretreatment comprised the physical abrasion of the shell surface and cleaning in deionized water within an ultrasonic bath to remove adhering material. The sample was then dried and crushed, and the outer 20% of the shell was removed by acid hydrolysis using 1M HCl. The last step was carried out immediately prior to acid hydrolysis of the remaining shell and CO<sub>2</sub> collection (cf. Vita-Finzi 1980; Heier-Nielsen et al. 1995).

All CO<sub>2</sub> samples were cryogenically purified and the gas split into 3 sub-samples. One sample was converted to graphite by the method of Slota et al. (1987) for accelerator mass spectrometer (AMS) analysis. <sup>14</sup>C/<sup>13</sup>C ratios were measured at the SUERC AMS facility (NEC 5-MV terminal voltage instrument operated at 4.5 MV with carbon in the 4+ charge state). The second sub-sample of CO<sub>2</sub> was used for δ<sup>13</sup>C analysis. The isotopic composition of the CO<sub>2</sub> was measured on a VG SIRA 10 stable isotope mass spectrometer using NBS standards 22 (oil) and 19 (marble) to determine the 45/44 and 46/44 atomic mass ratios, from which a sample δ<sup>13</sup>C value could be calculated. The third sub-sample was archived for possible future analysis.

All mollusk shell measurements were made on the same sample wheel to minimize variation resulting from measurement processes. One shell (201-02J) was measured twice in this wheel to assess any difference in <sup>14</sup>C age that resulted from the analysis of material from the inner and outer portions of a single mussel shell.

To examine the variability in mollusk shell <sup>14</sup>C age, the ages for a single species were compared using a  $\chi^2$  test (cf. Ward and Wilson 1978). The test assesses whether the internal variability of a group of measurements is consistent with the errors on the individual determinations. The test statistic ( $t$ ) was compared with the critical value for 95% significance ( $\chi^2_{:0.05}$ ) for the appropriate number of samples ( $n$ ) in a tested group. Where a group of 4 analyses did not pass the  $\chi^2$  test, the outlying data point was removed and the test repeated. <sup>14</sup>C measurements made on a single mollusk species that were statistically indistinguishable on the basis of the  $\chi^2$  test were combined to produce a weighted mean <sup>14</sup>C age for each species. The weighted mean ages were then examined to determine whether there was any between-species variability. Values of ΔR were calculated using all measurements that passed the  $\chi^2$  test for each mollusk species. Each ΔR value was produced by converting a terrestrial <sup>14</sup>C age ±1 σ to upper and lower 1-σ modeled marine <sup>14</sup>C age bounds using a linear interpolation of the measured atmospheric and modeled marine calibration curve data (Stuiver and Braziunas 1993). ΔR was then the difference between the midpoint of the modeled marine <sup>14</sup>C age bounds and the measured marine <sup>14</sup>C age, with an associated error derived from the model age bounds and the error on the measured marine age (Reimer et al. 2002).

To provide an empirical assessment of the variation in ΔR, we considered all possible marine/terrestrial pairs within the context and thus computed all possible values (maximum of 16) of ΔR for each marine species. Such a sensitivity study provides a measure of the robustness of the ΔR estimate to the arbitrary matching of the different samples from the same horizon. This distribution of ΔR values was then summarized using a weighted mean ΔR value and appropriate standard deviation. Two

approaches to estimation of the standard deviation are considered: the first takes the observed sample standard deviation around the weighted mean, accounting for quoted errors (“between”), while the second estimate is based only on the measurement quoted errors (“within”). The larger of the “within and between” sample standard deviations were chosen to reflect any additional sources of variation beyond that expected, given the quoted errors. The weighted mean  $\Delta R \pm 1 \sigma$  for each shell species was then compared using the  $\chi^2$  test to assess whether any significant difference in calculated value existed between the species.

**RESULTS**

The results for all  $^{14}\text{C}$  analyses are shown in Table 1. The results of the  $\chi^2$  analyses of the data are presented in Table 2, while weighted mean ages and  $\Delta R$  values for each species are presented in Table 3.

Table 1  $^{14}\text{C}$  and  $\delta^{13}\text{C}$  results for all samples measured during this study.

Measurement ID	Sample ID	Species	Age BP	$\delta^{13}\text{C}$
SUERC-93	201-01A	<i>Hordeum</i> sp.	2155 ± 40	-24.2
SUERC-94	201-01B	<i>Hordeum</i> sp.	2120 ± 40	-22.6
SUERC-95	201-01C	<i>Hordeum</i> sp.	2135 ± 40	-22.8
SUERC-96	201-01D	<i>Hordeum</i> sp.	2110 ± 80	-24.5
SUERC-3208	201-02Q	<i>Patella vulgata</i>	2435 ± 45	0.3
SUERC-3209	201-02R	<i>Patella vulgata</i>	2485 ± 45	1.9
SUERC-4113	201-02S	<i>Patella vulgata</i>	2410 ± 35	1.4
SUERC-3211	201-02T	<i>Patella vulgata</i>	2335 ± 35	1.1
SUERC-3196	201-02I	<i>Mytilus edulis</i>	2440 ± 35	1.3
SUERC-3197	201-02J	<i>Mytilus edulis</i>	2453 ± 43	1.2
SUERC-3199	201-02K	<i>Mytilus edulis</i>	2395 ± 35	1.6
SUERC-3200	201-02L	<i>Mytilus edulis</i>	2475 ± 35	0.6
SUERC-3201	201-02M	<i>Littorina littorea</i>	2400 ± 35	2.2
SUERC-3202	201-02N	<i>Littorina littorea</i>	2390 ± 35	2.4
SUERC-4123	201-02O	<i>Littorina littorea</i>	2415 ± 35	1.6
SUERC-3207	201-02P	<i>Littorina littorea</i>	2585 ± 35	2.1
SUERC-3212	201-02U	<i>Ensis ensis</i>	2520 ± 35	-0.4
SUERC-3216	201-02V	<i>Ensis ensis</i>	2455 ± 35	-0.1
SUERC-3217	201-02W	<i>Ensis ensis</i>	2425 ± 35	0.5
SUERC-3219	201-02X	<i>Ensis ensis</i>	2370 ± 35	0.4
SUERC-3220	201-02Y	<i>Cestroderma edule</i>	2505 ± 35	2.4
SUERC-3221	201-02Z	<i>Cestroderma edule</i>	2440 ± 35	2.3
SUERC-3222	201-02A	<i>Cestroderma edule</i>	2420 ± 40	0.6

Table 2 Mean age ±1 standard deviation and *t* values for the 6 species measured.

Species	Mean $^{14}\text{C}$ age ±1 std dev <sup>a</sup>	<i>t</i> value for species group
<i>Hordeum</i> sp.	2135 ± 22	0.49 ( $\chi^2_{:0.05} = 7.81$ )
<i>Patella vulgata</i>	2405 ± 31	7.63 ( $\chi^2_{:0.05} = 7.81$ )
<i>Mytilus edulis</i>	2440 ± 18	2.74 ( $\chi^2_{:0.05} = 7.81$ )
<i>Littorina littorea</i>	2448 ± 46	20.84 ( $\chi^2_{:0.05} = 7.81$ )
<i>Ensis ensis</i>	2443 ± 31	9.57 ( $\chi^2_{:0.05} = 7.81$ )
<i>Cestroderma edule</i>	2458 ± 26	2.97 ( $\chi^2_{:0.05} = 5.99$ )

<sup>a</sup>All age measurements included in calculations.

Table 3 Weighted mean age (excluding 2 outliers) and  $\Delta R$  values for the 5 mollusk species.

Species	Weighted mean $^{14}\text{C}$ age $\pm 1$ std dev	$\Delta R$
<i>Patella vulgata</i>	2405 $\pm$ 31	-74 $\pm$ 20
<i>Mytilus edulis</i>	2440 $\pm$ 18	-47 $\pm$ 20
<i>Littorina littorea</i>	2402 $\pm$ 20	-85 $\pm$ 22
<i>Ensis ensis</i>	2417 $\pm$ 25	-71 $\pm$ 23
<i>Cestroderma edule</i>	2458 $\pm$ 26	-32 $\pm$ 23

There was no significant difference in age between the inner and outer portions of shell 201-02J; therefore, the 2 measurements were combined to give a weighted mean age. The  $\delta^{13}\text{C}$  values were also combined. These are the values presented in Table 1.

Within the groups of measurements of *Littorina littorea* and *Ensis ensis*, there were significant differences in  $^{14}\text{C}$  age, as shown by the larger standard deviations and the calculated values of  $t$ . In both cases, the higher  $t$  statistics were mainly derived from single measurements. In the group of 4 measurements of *Littorina littorea* shells, SUERC-3207 is significantly older than the other measurements and responsible for the high  $t$  value. Similarly, in the group of *Ensis ensis* measurements, SUERC-3212 is older than the remaining measurements of this species. These outlying ages may represent: (i) material of an older age that was incorporated into the deposit during formation; (ii) material of the same age but of different activity—e.g. in the case of *Littorina*, which can occupy a range from the high shoreline to the sub-littoral fringe, the older age may derive from an individual collected from the sub-littoral fringe and the 3 younger samples from individuals collected near the high-water mark. The former would be feeding on algae rarely exposed to the atmosphere and the latter feeding on algae frequently exposed to the atmosphere. Cook et al. (2004) have demonstrated such differences in both winkles and limpets, although the data presented here show no evidence of this in the limpet shells that were analyzed; or (iii) they may represent measurement variability.

If SUERC-3207 and SUERC-3212 are excluded from the measurement groups, the values of  $t$  for these groups become  $t = 0.26$  ( $\chi^2_{:0.05} = 5.99$ ) and  $t = 3.03$  ( $\chi^2_{:0.05} = 5.99$ ), respectively, indicating that the other measurements were indistinguishable at 95% significance.

For each species, measurements that were statistically indistinguishable were combined to produce a weighted mean age (Table 3). When the weighted mean ages for the 5 mollusk species were compared, no significant differences were observed. The  $t$  value of the 5 weighted mean ages was  $t = 4.15$  ( $\chi^2_{:0.05} = 9.49$ ). Similarly,  $t = 22.22$  ( $\chi^2_{:0.05} = 26.3$ ) for the entire group of ages, excluding SUERC-3207 and SUERC-3212.

The calculated values of  $\Delta R$  for the different species are also detailed in Table 3, and again, a  $\chi^2$  test indicates no significant difference in  $\Delta R$  value ( $t = 3.87$  [ $\chi^2_{:0.05} = 9.49$ ]).

## DISCUSSION AND CONCLUSIONS

Environmental differences between the 5 mollusk species include shore position, food source, and habitat. Common limpets (*Patella vulgata*) and common winkles (*Littorina littorea*) are epifaunal grazers that inhabit hard substratum (limpets) and seaweed communities (winkles) from the high shore to the sub-littoral fringe. The limpets are microphagous grazers, subsisting upon the microalgal films (predominantly organic material, diatoms, and cyanobacteria) that coat rocky shores (Jenkins and Hartnoll 2001). Razor shells and cockles (*Ensis ensis* and *Cestroderma edule*, respec-



tively) are infaunal and burrow into soft sediment. These are active suspension feeders on organic debris in the water column. Cockles are found in the lower intertidal to subtidal zone, while razor shells inhabit extreme low water to the shallow sublittoral zone. Finally, *Mytilus edulis* (mussels) are active suspension feeders on phytoplankton, bacteria, detritus, and dissolved organic matter (DOM), and are found from the high intertidal to the shallow subtidal zone attached to the surfaces of rocks and other hard inorganic substrata.

Despite the differences in food sources and the ecological niches that the 5 mollusk species occupy, the variation in measured  $^{14}\text{C}$  ages from a single secure archaeological context does not exceed that which would be expected to result from measurement variability alone. The main conclusion that can be drawn from these results is that at Hornish Point, where there are no large-scale sources of carbon that may be selectively incorporated into specific mollusk species (e.g. the presence of carboniferous rocks or a significant freshwater input), no observable species-dependent variations in  $^{14}\text{C}$  age were observed. This indicates that differences in habitat and feeding behavior between the species that were studied do not have a significant influence upon the  $^{14}\text{C}$  activity of precipitated shell carbonate. The assessments of  $\Delta R$  made with the various mollusk species used in this study are therefore comparable, and no correction for species-dependent variation is required. It is likely that these conclusions can be extended to other sites of a similar nature.

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