# INFLUENCE OF MOLLUSC SPECIES ON MARINE ΔR DETERMINATIONS

Philippa L Ascough[1,2], Gordon T Cook[2], Andrew J Dugmore[1], E Marian Scott[3] and Stewart PHT Freeman[2]
[1]Institute of Geography, School of GeoSciences, University of Edinburgh, Edinburgh EH8 9XP, UK.

[2]Scottish Universities Environmental Research Centre, Scottish Enterprise Technology Park, Rankine Avenue, East Kilbride, Glasgow, G75 OQF, UK.
[3]Department of Statistics, University of Glasgow, Glasgow G12 8QQ, UK.

## Abstract

$^{14}C$ ages were measured on replicate samples of burnt grain and five mollusc 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 mollusc species on ΔR determinations that are calculated using the paired terrestrial/marine sample approach. The mollusc species examined inhabit a range of environments and utilise a variety of food sources within the intertidal zone. Several authors have suggested that these factors may be responsible for observed variations in the $^{14}C$ activity of mollusc shells that were contemporaneous in a single location. This study found no significant variation in the $^{14}C$ ages of the mollusc species, and consequently, no significant variation in calculated values of ΔR. The implication is that in an area where there are no carboniferous rocks or significant local input of terrestrial-source carbonate to the surface ocean water, any of a range of marine mollusc species can be used in combination with short-lived terrestrial material from the same secure archaeological context to accurately determine a ΔR value for a particular geographic location and period in time.

## Introduction

Marine mollusc 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 $^{18}O/^{16}O$ 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. $^{14}C$ age measurements made on mollusc shells are often used to give a chronological context to these records and this also enables comparisons to be made between different datasets. In addition to being a resource for paleoenvironmental data, marine mollusc shells are abundant in many regions of coastal archaeological remains and $^{14}C$ age measurements may be made on them when there is an absence of secure, dateable terrestrial material. A major consideration when using these $^{14}C$ measurements to establish a chronological framework for an archaeological site is the $^{14}C$ depletion of the oceans relative to the terrestrial biosphere – the so-called marine reservoir effect (MRE).

This 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 $CO_2$ exchange into river water. The effect is amplified in areas of enclosed coastal topography (*eg*. fjords) where circulation with the open ocean is limited and is reflected in shell carbonates precipitated in affected areas (Heier-Neilsen *et al.*, 1995). Mollusc shell $^{14}C$ activity can also be modified by exchange with environmental carbon (e.g. carbonates within percolating groundwater (Bezerra *et al.*, 2000)) during post-mortem recrystallisation. Thus, rigorous inspection and pre-treatment is required to avoid contaminated samples.

It is well established that the oceans are depleted in $^{14}C$ relative to the atmosphere and that this depletion varies both spatially and temporally. This depletion is translated to organisms that incorporate marine carbon, including molluscs. The carbonate shell of these organisms contains carbon derived from the dissolved inorganic carbon component (DIC) of the surrounding water mass, and its specific $^{14}C$ 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 regions. This removes a water mass from the atmosphere-ocean interface, and therefore prevents further gaseous exchange of $CO_2$ with the atmosphere. Such a water mass may then be resident in the deep oceans for an extended period, during which time its $^{14}C$ activity decreases due to radioactive decay. Upwelling eventually returns deep water to the surface ocean, which in turn depletes the surface water $^{14}C$ activity. The consequence of a MRE is that $^{14}C$ 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 dataset (Hughen *et al.*, 2004), which provides an average time-dependant correction for the age of the global surface ocean. The deviation of marine $^{14}C$ 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.*, 2004). 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 mollusc shells are often used in determination of $\Delta R$. 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-dependant variations in shell $^{14}C$ activity mean that significantly different MREs (and therefore $\Delta R$ values) can be derived from measurements made on various mollusc species at a single location (Forman and Polyak, 1997; Hogg *et al.,* 1998). In contrast, Harkness (1983) found no clear species-dependant variations in natural enrichment in an assessment of modern UK coastal MRE. Different marine mollusc species inhabit a range of ecological niches within the intertidal zone and deeper ocean where a variety of feeding mechanisms and food sources are utilised. These differences are commonly identified as responsible for observed inter-species variation in shell $^{14}C$ activity. For example, Ingram (1996) suggests that species-dependant variations in mollusc shell $^{14}C$ 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 $CaCO_3$ has a different $^{14}C$ activity to that of other species this would result in different calculated ΔR 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. Mollusc habitats include the hard substrate (*eg*. bedrock outcrops), the sediment surface (epifaunal position) and below the sediment surface (infaunal position), while feeding mechanisms include grazing upon micro-algae, 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 utilised by filter feeders usually has a $^{14}C$ activity that is closer to ocean DIC and may mean that filter feeders, (*eg*. mussels and oysters) incorporate proportionally lower amounts of atmospheric $^{14}C$ than herbivorous grazing species (*eg*. 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 mollusc during grazing (Dye, 1994), or while inhabiting carbonate-rich sediments (Forman and Polyak, 1997). Dyke *et al.* (2002) suggest that the elevated $^{14}C$ age of the deposit-feeding marine mollusc *Portlandia arctica* is the result of its infaunal position and feeding mechanism, while Forman and Polyak (1997) observed that molluscs with sessile habitats and pelagic food sources gave significantly lower MRE offsets (*ie*. a younger $^{14}C$ age).

This paper examines the variation in $^{14}C$ age and values of ΔR that can be derived from mollusc shells of five 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 (Fig 1), in an area where there is no significant local input of terrestrial-source carbonate to the surface ocean water.

## Methods

Samples of carbonised cereal grain and marine mollusc 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 carbonised grain and marine mollusc shells of several species, making it suitable for the paired sample approach to determining ΔR.

Four individual carbonised barley (*Hordeum sp.*) grains were taken for $^{14}C$ measurement, together with five different species of mollusc 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 three 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 pre-treatment 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 pre-treatment, the shells were inspected and only non-porous specimens with preserved textures were selected for analysis (Mangerud, 1972, Mook and Waterbolk, 1985). Pre-treatment comprised the physical abrasion of the shell surface and cleaning in deionised water within an ultrasonic bath to remove adhering material. The sample was then dried and crushed followed by removal of the outer 20% of the shell by acid hydrolysis using 1M HCl. The last step was carried out immediately prior to acid hydrolysis of the remaining shell, and $CO_2$ collection (c.f. Vita-Finzi, 1980; Heier-Nielsen *et al.,* 1995).

All $CO_2$ 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 AMS analysis. $^{14}C/^{13}C$ ratios were measured on 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 $CO_2$ was used for $\delta^{13}C$ analysis. The isotopic composition of the $CO_2$ 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 $\delta^{13}C$ value could be calculated. The third sub-sample was archived for possible future analysis.

All mollusc 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 $^{14}C$ age that resulted from the analysis of material from the inner and outer portions of a single mussel shell.

To examine the variability in mollusc shell $^{14}C$ 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. $^{14}C$ measurements made on a single mollusc species that were statistically indistinguishable on the basis of the $\chi^2$ test were combined to produce a weighted mean $^{14}C$ age for each species. The weighted mean ages were then examined to determine whether there was any between-species variability. Values of $\Delta R$ were calculated using all measurements that passed the $\chi^2$ test for each mollusc species. Each $\Delta R$ value was produced by converting a terrestrial $^{14}C$ age $\pm$ 1$\sigma$ to upper and lower 1$\sigma$ modelled marine $^{14}C$ age bounds, using a linear interpolation of the measured atmospheric and modelled marine calibration curve data (Stuiver and Braziunas, 1993). $\Delta R$ was then the difference between the midpoint of the modelled marine $^{14}C$ age bounds and the measured marine $^{14}C$ 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 summarised 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 $^{14}C$ 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 ΔR values for each species are presented in Table 3.

There was no significant difference in age between the inner and outer portions of shell 201-02J, therefore, the two measurements were combined to give a weighted mean age. The $\delta^{13}C$ 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 $^{14}C$ 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 four 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 eg. 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 analysed, 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 five mollusc species were compared, no significant differences were observed. The *T*-value of the five 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 ΔR for the different species are also detailed in Table 3 and again, a $\chi^2$ test indicates no significant difference in ΔR value ($T = 3.87$ ($\chi^2_{:0.05} = 9.49$)).

## Discussion and Conclusions

Environmental differences between the five mollusc 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*, respectively) 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 five mollusc species occupy, the variation in measured $^{14}C$ 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 mollusc species (*eg*. the presence of carboniferous rocks or a significant freshwater input), no observable species-dependant variations in $^{14}C$ age were observed. This indicates that differences in habitat and feeding behaviour between the species that were studied do not have a significant influence upon the $^{14}C$ activity of precipitated shell carbonate. The assessments of ΔR made with the various mollusc species used in this study are therefore comparable, and no correction for species-dependant variation is required. It is likely that these conclusions can be extended to other sites of a similar nature.

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

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

**Table 2** Mean age ± 1standard deviation and *T* –values for the six species measured.

| Species | Mean $^{14}C$ age ± 1std dev* | *T* value for species group |
|---|---|---|
| *Hordeum sp.* | 2130 ± 17 | 0.49 ($\chi^2_{:0.05} = 7.81$) |
| *Patella vulgata* | 2416 ± 54 | 7.63 ($\chi^2_{:0.05} = 7.81$) |
| *Mytilus edulis* | 2441 ± 29 | 2.74 ($\chi^2_{:0.05} = 7.81$) |
| *Littorina littorea* | 2448 ± 80 | 20.84 ($\chi^2_{:0.05} = 7.81$) |
| *Ensis ensis* | 2443 ± 54 | 9.57 ($\chi^2_{:0.05} = 7.81$) |
| *Cestroderma edule* | 2455 ± 36 | 2.97 ($\chi^2_{:0.05} = 5.99$) |

* all age measurements included in calculations

**Table 3** Weighted mean age (excluding 2 outliers) and ΔR values for the five mollusc species.

| Species | Weighted mean $^{14}C$ age ± 1 std dev | ΔR |
|---|---|---|
| *Patella vulgata* | 2405 ± 31 | -74 ± 20 |
| *Mytilus edulis* | 2440 ± 18 | -47 ± 20 |
| *Littorina littorea* | 2402 ± 20 | -85 ± 22 |
| *Ensis ensis* | 2417 ± 25 | -71 ± 23 |
| *Cestroderma edule* | 2458 ± 26 | -32 ± 23 |

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