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Best Practice Methodology for ^{14}C Calibration of Marine and Mixed Terrestrial/Marine Samples

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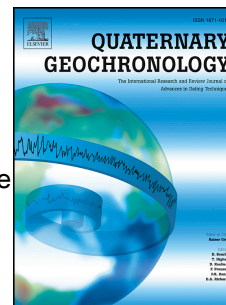
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Best Practice Methodology for ^{14}C Calibration of Marine and Mixed Terrestrial/Marine Samples

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Abstract

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Introduction

In archaeology, the ideal situation when employing radiocarbon dating as the chronological tool is where: 1. The archaeological contexts that can provide the samples necessary to answer the dating questions are sealed. 2. These contexts were formed (and sealed) over short periods of time. 3. The sample taphonomy is well understood and there is no question of the samples being residual, and 4. Terrestrial samples (preferably short-lived), suitable for dating, are available within these contexts e.g. burnt cereal grain, round-wood charcoal, articulated terrestrial herbivore bones (articulated human bones with terrestrial stable isotope dietary signals), etc. Under such circumstances, radiocarbon dating and subsequent calibration using programs such as OxCal, Calib, etc. is relatively straightforward. Furthermore, where a well-designed programme of sample selection has been undertaken, taking into account the site stratigraphy and phasing, Bayesian analysis may increase the chronological precision that can be applied to the dating questions. However, the question arises of "What is the best way forward when marine-influenced samples are the only datable materials present?" These would include wholly marine samples (e.g. fish bones, mollusc shells) or partially marine samples (e.g. human remains from someone with a partial marine diet).

Calibration of Marine Samples

Historically, the treatment of $^{14}\text{C}/^{12}\text{C}$ measurements made on marine samples (mainly shell) has been variable in approach. Harkness (1983) noted that the problem centred on the

method (if indeed any was employed) used to normalise the $^{14}\text{C}/^{12}\text{C}$ ratio for isotopic fractionation prior to age calculation. At that time, laboratories employed one of the three following approaches: 1. No fractionation correction was applied, i.e. ages were calculated on the basis of the measured $^{14}\text{C}/^{12}\text{C}$ ratio. 2. Normalisation was applied relative to a $\delta^{13}\text{C}$ (V-PDB) value of 0‰ (mean $^{13}\text{C}/^{12}\text{C}$ enrichment value for marine carbonate) or 3. Normalisation was applied relative to a $\delta^{13}\text{C}$ (V-PDB) value of -25 ‰ (mean $^{13}\text{C}/^{12}\text{C}$ enrichment value for wood), which is the conventional method of age calculation. In the first two cases, this results in radiocarbon ages that are approximately 400 years younger than if approach 3 were employed and of course, the 400 years approximates to the global average surface water marine reservoir effect (MRE), making it a fortuitous compensation for this effect. This 400-year offset also approximates to the apparent MRE around the UK coastline (405 ± 40 years) as estimated by Harkness (1983). This has resulted in a serious misunderstanding of the MRE by many archaeologists and of course, this apparent 400-year compensation would be at odds with the effect in other geographical areas such as where upwelling of 'old' water occurs (e.g. Soares & Dias 2006).

A significant advance in dealing with marine samples was made by Stuiver et al. (1986) who defined the reservoir age as $R(t)$, which is the difference in conventional ^{14}C age between contemporaneous samples from the atmospheric and marine environments. They derived a separate (model) calibration curve, using the atmospheric $\Delta^{14}\text{C}$ data, that incorporated time-dependant variability in $R(t)$, back to 9,000 cal years BP (The current marine calibration curve (Marine 13) (Reimer et al. 2013) extends to 50,000 cal years BP). In addition to a temporally variable $R(t)$, there are regional deviations from the contemporaneous global average $R(t)$ value, which are a function of climate and oceanic circulation systems and are expressed as ΔR values. Thus, for the most accurate calibration of ^{14}C age measurements on marine samples, a ΔR value for the geographic region and time period of interest is required. Derivation of this ΔR can be accomplished by a number of techniques including:

1. Obtaining samples of marine organisms (generally marine mollusc shells) from a precisely known location and where the pre-1950 calendar date of collection was recorded. The difference in ^{14}C activity between the marine sample and the global atmosphere (from the global atmospheric calibration curve) for the year of sample collection is used to calculate the 'apparent age' of the material, which, in turn, allows determination of the local deviation from the global $R(t)$ (Mangerud and Gulliksen, 1975; Harkness, 1983; Olsson, 1980; Birkenmajer and Olsson, 1998). The disadvantages of this technique are two-fold: (i) the samples are typically of recent origin (i.e. < 200 years old) and so longer-term temporal studies of MRE variability are not possible and (ii) multiple samples are often lacking and hence only single calculations can be performed.

2. Using tephra isochrones: In terms of the ^{14}C timescale, the deposition of tephra from a volcanic eruption is instantaneous. The technique relies on ^{14}C analysis of samples associated with a tephra layer found within onshore ice cores or other accumulating deposits such as lake sediments or peat profiles and within offshore accumulating marine sediments (e.g. Austin et al., 1995; Hafliðason *et al.*, 2000; Thornalley et al., 2011). The analysis of samples such as foraminifera associated with the marine sediment and plant macrofossils or some other fraction of the peat/sediment allows calculation of the ΔR value by conversion of the terrestrial ^{14}C age to a modelled marine age; the difference between this and the

conventional ^{14}C age of the marine sample represents the ΔR . The disadvantages of this technique are: (i) ΔR calculations are necessarily confined to periods in time when volcanic eruptions occurred and where the tephra were deposited and (ii) in marine sediments in particular there is the possibility of slow accumulation in parallel with significant mixing of surface sediments by bioturbation.

3. Using paired marine and terrestrial samples, typically from secure contexts within coastal archaeological sites or from coastal deposits resulting from tsunamis, etc. (Southon et al., 1992; Kennett et al., 1997; Ascough et al., 2004; Russell et al., 2010). This often takes the form of measuring the ^{14}C ages of single pairs of samples (Kennett et al., 1997). Again, the terrestrial ^{14}C age is converted to a modelled marine age and the difference between this and the conventional ^{14}C age of the marine sample represents the ΔR . The advantage of a multiple pair approach, as advocated by Ascough et al. (2009) and Russell et al. (2010), is that an anomalous ^{14}C age in an otherwise coherent body of measurements can be identified and would be indicative of intrusive or re-worked material. A simple chi-squared (χ^2) test of the marine and terrestrial data can be employed to demonstrate that the ages are indicative of a single deposition time (within statistical limits). Short-lived terrestrial materials such as round-wood charcoal, carbonized cereal grains or terrestrial herbivore bones are suitable for dating while the shells of short-lived mollusc species are ideal marine samples. The disadvantages of this approach are the spatial and temporal limitations of the archaeological sites or tsunami events.

4. Sclerochronology: This is the equivalent of dendrochronology using cross-matching of annually banded, long-lived bivalves such as *Arctica islandica* (Marchitto et al. 2000; Scourse et al. 2006). Butler et al. (2009) have used sclerochronology to determine variations in ΔR over the past 500 years on *A. islandica* from the south and west of the Isle of Man (off the west coast of mainland UK). In a similar manner, Helama and Hood (2011) use anthropogenically-deposited *A. Islandica* excavated from a Stone Age midden in N. Norway to yield a 155-year multi-shell sclerochronology from which ΔR values could be determined. The advantage of this technique is the ability to determine annual ΔR values in absolute time, while the disadvantages are that the chronologies will be geographically limited to where *A. islandica* are found while the temporal range of the chronologies will be limited by the age of shells that are recovered for chronology construction.

The methods discussed above often employ single samples/single pairs of marine/terrestrial samples and this can result in significant problems. We selected one example from the scientific literature (Kennett et al., 1997) where the authors were using the single paired-sample approach. In one context they measured the ^{14}C age of a charred twig that produced a ^{14}C age of 6000 ± 70 years BP and a mussel shell that gave a ^{14}C age of 6500 ± 80 years BP. The equivalent marine model age of the terrestrial sample was determined by the authors to be 6420 ± 70 years BP and hence the ΔR value for this sample pair was 80 years. We then randomly generated 4 terrestrial ^{14}C ages that are statistically indistinguishable from the measured age of 6000 ± 70 years BP and 4 marine ages that are indistinguishable from the marine age of 6500 ± 80 years BP. From these, we generated ΔR values for all 16 possible marine/terrestrial combinations as shown in Table 1.

The results in Table 1 illustrate a wide range of ΔR values from -253 to $+261$ years, indicating that the single value of 80 as calculated on a single pair of measurements has the

potential to be totally misleading when determining a ΔR value for a particular region and period. The average ΔR was 16 years, and the standard deviation was 136 years, while the standard error on the mean was 34 years. The magnitude of the standard deviation captures the nature of the variability within the population of possible matched samples.

The approach adopted at SUERC is to use the multiple marine and terrestrial sample approach (typically four samples of each), thereby generating 16 possible ΔR values when all samples pass their respective χ^2 test. This work has resulted in a number of publications in which we have quoted mean ΔR values with their associated standard error term (Ascough et al. 2005a, 2005b, 2006, 2009; Russell et al. 2010). However, we have recently questioned our method of using the standard error to provide a measure of the variability that would encompass any **future individual measurements of ΔR** made on a single pair of samples from the same context (Russell et al. 2011a, 2011b). The standard error on the mean represents how precisely we know the mean value of a population. However, if we wish to make comment about a future hypothetical ΔR value calculated from this population, we also need to include a measure of the variability within that population, i.e. the standard deviation. This is highlighted in Table 1 as discussed above and was also highlighted in our own research on ΔR for a Medieval archaeological site (Archerfield) situated on the east coast of Scotland (Russell et al. 2011a). Here, for a single context, a ΔR value of -42 ± 5 (1σ) years was determined, based on ^{14}C analysis of 8 terrestrial and 8 marine samples (64 ΔR values from all possible pairings). However, the individual values varied from +34 to -122 and so, the standard error of 5 years does not reflect the variability in the ΔR values. In light of this, it was proposed that the standard error for predicted values should be used; this is calculated by:

$$\sigma = \sqrt{(x^2 + y^2)}$$

where x is the error on the weighted mean and y is the standard deviation on the ΔR values. On this basis we calculated a weighted mean ΔR value for Archerfield of -33 and a standard error for predicted values of 43 ($n = 64$). Similarly, for Table 1, the mean ΔR value is at odds with the single value of +80 ^{14}C yrs originally calculated by Kennett et al. (1997) and the standard error of ± 9 that we calculated fails to describe the potential variability in ΔR values. The standard error for predicted values, using the data in Table 1, is 140 years ($n=16$) and although this will have the effect of increasing the calibrated age range, its use should lead to a more accurate (if less precise calibrated calendar age range). The prediction error represents the uncertainty on a single future observation of ΔR from the population represented by previous observations of ΔR and their observed scatter. Based on the ΔR values estimated, the prediction error can be used to calculate a confidence interval within which a single future ΔR value is likely to lie, with some level of confidence. This would be used where we could be dealing with a similar (geographical) site, but which does not have suitable samples, or where there is only a single sample (or one pair of samples).

Therefore, for the most accurate method of calibrating marine samples we would recommend:

1. Using the most recent marine curve – Marine13.

2. Using a ΔR value appropriate for the geographic area and time period of interest. We would suggest that a value derived from a single pair of samples is potentially misleading and that a mean ΔR value based on several pairs of data from a single context should be employed or else a mean value based on a number of studies for the particular area and period of interest. In the absence of an appropriate ΔR value for the region and period, an appropriate regional value should be used. In a UK context, Reimer et al. (2002) give a value of -33 ± 93 for Ireland, Scotland and the Orkney Islands based on 31 sample pairs from 14 sites. Our own regional value (using data from the Neolithic through to the Late Medieval period) gives a value of -47 ± 52 (Russell et al. unpublished). Table 2 demonstrates how the calibrated age range for a radiocarbon age measurement of 2320 ± 20 years BP changes according to the method of calibration. Figure 1 illustrates the changes that occur in calibrated age ranges through the use of different calibration procedures. The overall effect of using the best possible estimates of the local ΔR value is a likely loss in precision but we would propose that the calibrated age range will be a more accurate reflection of the true age.
3. Ensure that the error on the ΔR value is appropriate; the standard error for predicted values is preferable in the situations described above.

Calibration of Mixed Marine/Terrestrial Samples

Calibration becomes even more complex when dealing with samples that contain both terrestrial- and marine-derived carbon. Here, the most frequently measured sample type is bones from inhumations, but can also include non-human omnivores, such as pigs and dogs that are eating marine resources. Typically, percentage marine diet is calculated from a linear plot of human bone collagen $\delta^{13}\text{C}$ values between terrestrial and marine diet end-member values. One of the most credible examples of this is the study of a change in diet of the Greenland Vikings (Arneborg et al., 1999). They used terrestrial and marine end-member values of -21.0‰ and -12.5‰ , respectively, when calculating the marine dietary component of Greenland Vikings who died between the late-10th and mid-15th centuries. The terrestrial end-member value was based on values derived from inland Swedish populations ranging through the Viking and Medieval periods to a 17th century population, while the marine end-member value was based on a study of Thule Culture Eskimos who lived close to the Vikings in both time period and location. Hence, both end-members values are well founded. However, there are many other examples where estimation of the percentage marine diet would not be as straightforward and it is questionable how closely the contribution of marine resources to diet can be estimated given the following:

1. A fundamental assumption in most studies is that stable isotope ratios in human bone collagen only reflect the protein component of the diet; however, in low protein diets, such as might be found during the Neolithic, the carbon derives from all dietary components, i.e. fat, carbohydrates and protein (Ambrose and Norr, 1993). $\delta^{13}\text{C}$ values in carbohydrates and fat are depleted relative to the protein component, therefore a diet based on low protein plant food (cereals) would result in a relatively light $\delta^{13}\text{C}$ value (Hedges, 2004). Bonsall et al. (2009) demonstrated that a significant proportion of marine food (e.g. shellfish) could be incorporated into such a diet and still result in what appears to be a 'terrestrial' $\delta^{13}\text{C}$ signal.

Therefore, to make an informed judgement on human diet, stable isotope data for human bone collagen (i.e. $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$, and possibly $\delta^{34}\text{S}$ values) are required and in the absence of suitable populations who consumed dominantly terrestrial or marine resources to provide suitable $\delta^{13}\text{C}$ end-members, these stable isotope values should be viewed in conjunction with knowledge gained from associated archaeological (midden) deposits containing remains of the likely dietary components (animal & fish bones, marine shells, etc.). $\delta^{13}\text{C}$ values for marine shell cannot be related to the original soft tissues; however, flesh values can be obtained from the analysis of carefully selected modern analogues.

2. Hedges (2004) pointed out that a 1‰ change in the terrestrial end point corresponds approximately to a 10% change in the intake of marine resources. Therefore, a terrestrial end-point value with a $\pm 1\%$ uncertainty would be equivalent to a difference of 20% in terms of determining the minimum level of marine resource consumption. This highlights the need for relevant dietary end-members and to consider carefully any errors that should be applied in calculations of percentage marine diet.

3. Table 3 provides indicative values for a number of resources and indicates the variability in $\delta^{13}\text{C}$. The $\delta^{13}\text{C}$ values of the marine resources consumed can vary significantly and this can have a significant impact on any calculation of percentage marine diet, as Bonsall et al. (2009) observed. For example, assuming 4.5‰ enrichment from all food resources to human bone collagen (Hedges 2004), a hypothetical Neolithic diet was estimated to result in a $\delta^{13}\text{C}$ value of -20.9% . The authors demonstrated that the addition of 50% marine protein in the form of fish resulted in a $\delta^{13}\text{C}$ value of -16.6% , while the addition of 50% marine protein from oyster resulted in a value of -18.7% . On a linear plot of $\delta^{13}\text{C}$ values between terrestrial and marine end-members of -20.9% and -12.5% , respectively, this would result in percentage marine diets of 51% and 26%, respectively. This in turn would have a significant influence on the calibrated age range. Again, using the age of 2320 ± 20 BP, Marine 13 and a ΔR value of -47 ± 52 , a diet of $51 \pm 10\%$ marine resources would result in an age range of 360–120 cal BC while $26 \pm 10\%$ marine resources would result in an age range of 390–200 cal BC.

4. Stable isotopes provide information on an individual's diet, whereas, at best, archaeological data provide information on group diet, but are strongly affected by taphonomic and other biases. Plants will be under-represented in the stable isotope signal because of low protein and poor survival. This under-representation could also apply to fish bones because of inadequate sieving of deposit material during excavation, as well as poor survival. Also, in a pastoralist society, many of the resources (e.g. milk and cheese) will leave no direct trace except as lipids absorbed into pottery.

5. In some hunter-gatherer communities, sites are not necessarily occupied all year round, and important dietary resources may be consumed only at certain times of year and be entirely absent from the sites occupied during other seasons.

6. The lack of definitive information on the enrichment in $\delta^{13}\text{C}$ between the flesh of dietary resources and human bone collagen. The assumption is generally made that this is about 4.5 to 5‰ (e.g. van der Merwe and Vogel, 1978; Schoeninger, 1985; Hedges 2004), although smaller offsets (in the order of 2.5‰) between fish flesh and human bone collagen have been suggested (cf. Van der Merwe et al., 1993; Bonsall et al., 1997; Fischer et al.,

2007) (N.B. This 4.5–5‰ shift between the flesh of dietary resources and human bone collagen should not be confused with the approximate 1‰ shift between animal bone and human bone collagen).

Therefore, in the absence of suitable populations whose bone collagen $\delta^{13}\text{C}$ values can be used as analogues for the terrestrial and marine end members, their estimation is extremely complex, and while it may be impossible to determine accurate end-member values it should always be borne in mind that a sensible approach to the problem should always result in a more accurate calibrated age range. We recommend that the following methodology can be applied, which requires significant collaboration between the radiocarbon laboratory staff and the excavators:

1. Obtain as much information as possible on the likely dietary components from associated archaeological deposits through consultation with the excavator(s). This would include information on the presence of (i) terrestrial mammal bones, (ii) fish bones, (iii) shellfish remains and (iv) carbonized cereal grain, all of which would be indicative of resources consumed.

2. Estimate the approximate percentages of the various terrestrial resources in the diet through consultation with relevant archaeologists; undertake stable isotope analyses on the bone collagen and the protein component of any modern analogues that may be required. Determine a weighted mean terrestrial end-member $\delta^{13}\text{C}$ value for human bone collagen having taken into account (i) the likely proportions of each terrestrial component within the diet, (ii) an appropriate trophic enrichment of 4.5‰ between food resource and human bone collagen and (iii) a 1‰ enrichment between mammalian and human bone collagen (e.g. Post et al. 2002).

3. Where fish have been the dominant marine resource, repeat the above process to determine a marine $\delta^{13}\text{C}$ end member. If any estimation of the relevant proportions of fish and shellfish is possible: where shellfish have been the dominant dietary component, collect modern analogues from a suitable environment close to the original archaeological site and undertake $\delta^{13}\text{C}$ analysis of the protein component and calculate the marine end member using an enrichment to collagen of 2.5‰ (Van der Merwe et al., 1993; Bonsall et al., 1997). Where both fish and shellfish have been consumed, approximate proportions should be estimated and a marine end member $\delta^{13}\text{C}$ value calculated from them.

Obviously, much of the above can be subjective but again it should always be borne in mind that, given a sensible approach to the above, the resulting calibrated age range should be closer to the true age than a calibration that does not take marine diet into account. An example of this is our recent work on the remains of what were purported to be King Richard III. In August 2012, the University of Leicester began the search for the lost grave of King Richard III, in collaboration with the Richard III Society and Leicester City Council. It was thought that Richard, who was the last English king to die in battle, had been buried in the church of the Grey Friars in Leicester; however, any physical trace of the actual church had long been lost. Incredibly, the excavation very quickly uncovered both the friary and a skeleton with spinal curvature, consistent with descriptions of the king. A full discussion of the excavation strategy is provided in Buckley et al. (2013).

Richard III died at the battle of Bosworth (AD 1485), which was the last significant battle of the Wars of the Roses, the civil war between the Houses of Lancaster and York. Two samples of rib bone were received and prepared for high precision AMS radiocarbon dating and stable isotope analysis. In addition, the Oxford Radiocarbon Accelerator Unit also received 2 similar samples. The results are presented in Table 4. The four radiocarbon measurements are statistically consistent and were combined to form a weighted mean Greyfriars 2012 (451 ± 11 BP) (Hamilton and Bronk Ramsey, 2012). Calibration of this age gave a range of AD 1430–1460 (95.4% probability), which is significantly earlier than his year of death. However, both the $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ values for the four samples indicate that this individual had a varied, protein-rich diet that included non-terrestrial resources in the form of seafood. It is known that both oysters and marine fish were available and consumed by people across social classes in the medieval period but it was not possible to make any comment on the proportions of oyster and fish he consumed, other than that the heavy $\delta^{15}\text{N}$ value would tend to favour fish as the main marine component. In the absence of any means of determining dietary end-members, we chose -12.5‰ (purely marine) and -21‰ (purely terrestrial) as described by Arneborg et al. (1999). Using an average $\delta^{13}\text{C}$ value of -18.51‰ for the bone collagen samples, this equated to approximately 29% marine diet and in line with Hedges (2004) we placed a 10% error on this value. A ΔR value of -29 ± 51 for Medieval Britain (Russell 2011c) was employed. The calendar age range was then established using a mixed modelling approach to account for this percentage of marine protein. The result (cal AD 1460–1650) was then placed into a Bayesian statistical model, using the OxCal program (Version 4.2) (Bronk Ramsey 1995, 1998, 2001, 2009), to determine the most probable date of the sample given that the burial would have occurred prior to the Dissolution of Greyfriars (ca. AD 1538). The result (Fig. 2) indicates that the individual most probably died at some time in the period cal AD 1460–1540 (95.4% probability), consistent with the historical record of Richard's death in AD 1485. Subsequent comparison of mitochondrial DNA between a descendant on the maternal line and that of the remains provided confirmatory evidence that the remains were indeed those of Richard III (King et al. 2014). This demonstrates that a sensible calculation of percentage marine diet and subsequent calibration can yield improved results, even when the only available information is the stable isotope values from the remains.

A further example of calibration of human remains influenced by a marine diet comes from our recent research on the farmstead of Hofstaðir in north-east Iceland (Sayle et al. 2014). The farmstead lies on the River Laxá, 5 km west of Lake Mývatn, and has been documented as an area of major archaeological importance with respect to the settlement of Viking communities in Iceland (Friðriksson and Vésteinsson, 1997; Vésteinsson, 1998; Lucas and McGovern, 2007; McGovern et al., 2007; Lucas, 2009). The first human settlement of Iceland (*landnám*) is well documented to have occurred circa AD 870 (Sveinbjörnsdóttir et al. 2004). Tephrochronological studies indicate that the settlement of Hofstaðir occurred shortly after AD 940 but by the Hekla eruption of AD 1104, the site had been abandoned for approximately 70 years (Sigurgeirsson, 2001; Lucas, 2009).

In this case study, we make use of specific dietary and ΔR information. All of the 9 dated human remains shown in Table 5 come from what are regarded as the earliest two phases of the Hofstaðir cemetery (Gestsdóttir, pers. comm.). McGovern (pers. comm.), who has been very closely involved in excavating the site, indicated that the main terrestrial dietary

components were dairy products and domestic animal flesh while the main marine dietary component was dried fish. Therefore, the terrestrial dietary end member was based on the average $\delta^{13}\text{C}$ value for collagen extracted from herbivore bones excavated at Hofstaðir (N=30) ($\delta^{13}\text{C} = -21.7 \pm 0.5\text{‰}$) (Sayle et al. unpublished) (N.B. Sayle et al. (2014) used herbivore data from Skútustaðir). The marine end member was based on fish data (N=9) (average $\delta^{13}\text{C} = -14.3 \pm 0.3\text{‰}$) from the neighbouring site of Skútustaðir (Sayle et al., 2013). Taking into account the subsequent trophic level shift of *ca.* +1‰ that would occur between animal and human bone collagen (DeNiro and Epstein, 1978), we would expect that humans at Hofstaðir consuming a wholly terrestrial diet would display $\delta^{13}\text{C}$ values of approximately – 20.7‰, while those eating solely marine produce would have $\delta^{13}\text{C}$ values of approximately – 13.3‰. On this basis, the percentage marine diet of each individual was calculated from their $\delta^{13}\text{C}$ value using a linear interpolation between the 2 end-members. In addition, we re-calculated the mean ΔR value for this site and period and the associated standard error for predicted values ($\Delta\text{R} = 114 \pm 29$), based on the data given in Ascough et al. (2007b) (NB Sayle et al. (2014) used a regional ΔR value of 58 ± 75 , derived from the Queens University Marine Reservoir Correction Database). Using the derived value for percentage marine diet and the ΔR value of 114 ± 29 we re-calibrated all the age ranges using the mixed Marine 13/IntCal 13 curves, regardless of whether the dietary component was thought to be freshwater or marine,

Table 5 illustrates radiocarbon ages, calibrated age ranges (based on the assumption that the diets were entirely terrestrial), recalibrated age ranges (based on the assumption that the $\delta^{13}\text{C}$ values reflect the degree of marine resources in the diet – discussed fully below) and stable isotope values.

From the $\delta^{13}\text{C}$ values, Sayle et al. (2014) interpreted SK013, SK047 and SK053 as having almost exclusively terrestrial diets (denoted as T in Table 5). Our results suggest <10% marine resources for all three. Re-calibration for the small marine dietary component shifts the age ranges forward at the younger limit by 40–50 years. The age ranges are consistent, before and after correction for marine diet, with the established chronology for the occupation of Hofstaðir, namely that tephrochronological studies have demonstrated the settlement occurred shortly after AD 940 and was abandoned by around AD 1030. The authors interpreted the relatively high $\delta^{13}\text{C}$, $\delta^{15}\text{N}$ and $\delta^{34}\text{S}$ values for SK007, SK056 and SK009 as individuals with a significant marine component in their diets (denoted as M in Table 5). Recalibration shifts the age ranges of SK007 and SK056 to ones that are more in-keeping with the established chronology. SK009 has an acceptable age range with and without re-calibration for the marine dietary offset. Low $\delta^{34}\text{S}$ values accompanied by high $\delta^{13}\text{C}$ values in the human bone collagen of SK016, SK061 and SK066 were interpreted as being indicative of a significant freshwater component in the diet (denoted as F in Table 5). Despite recalibration of SK016, SK061 and SK066 for the marine dietary offset, they still had calibrated age ranges that were several centuries pre-*landnam*, highlighting the fact that there was a significant freshwater component in the diet of these individuals. Ascough et al. (2010) have demonstrated that freshwater resources from this area exhibit a reservoir effect in excess of 5000 ^{14}C years in some biota, hence the pre-*landnám* calibrated age ranges even following recalibration for the marine dietary offset. Ascough et al. (2010) have also shown that the freshwater reservoir effect is variable both spatially and temporally, which would most likely preclude re-calibration of these 3 samples.

While the examples we have provided follow the method of linear interpolation between two end members, new tools (i.e. FRUITS, Fernandes et al. 2014; SIAR, Parnell et al. 2010) that utilise a Bayesian approach to model the percent input from various food sources are becoming available. These approaches result in a probability density function that will hopefully provide a more accurate estimate for the percent protein dietary contribution and error from terrestrial, marine and freshwater resources. While SIAR only provides a two-isotope approach, FRUITS enables the use of three or more stable isotopes, or other isotopic data that might be useful, for estimating the palaeodietary inputs from the different carbon reservoirs. We are currently engaging with these new tools in an effort to provide a more robust approach to determining the various dietary components.

Conclusions

The calibration of marine samples requires the best possible estimate of a relevant ΔR value with a realistic error term that can be used as input terms to calibration programs such as OxCal and Calib. The most relevant ΔR value would be derived from the same geographic region and time period as the samples to be calibrated. We would not recommend using a value derived from a single pair of marine and terrestrial samples but rather a value based on multiple pairs with a standard error for predicted values. In the absence of such a ΔR value, a regional value such as can be obtained from the Marine Reservoir Correction Database held by Queens University Belfast (<http://calib.qub.ac.uk/marine/>) would be suitable. For human remains where there is clear evidence of a significant marine component, the same argument for a ΔR value and associated error term applies. We would recommend estimating the percentage marine diet from a linear plot of $\delta^{13}\text{C}$ values between terrestrial and marine end-members derived from suitable populations and where this is not feasible, to estimate these end-members based on stable isotope analysis of associated food remains from midden deposits. Here, detailed discussions with the relevant excavators are vital in obtaining the best possible estimates of the importance of different dietary components. It is important that a suitable error is placed on the estimate of the percentage marine diet component, and in line with Hedges (2004), we would recommend 10%. Also, the type of marine resource consumed (fish versus certain shellfish) is important in terms of deriving a suitable marine end-member as many shellfish species have significantly lower $\delta^{13}\text{C}$ values than fish. Currently, we are actively engaging with FRUITS to provide as robust an approach as possible to determining dietary components and associated errors from different reservoirs.

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Terrestrial ¹⁴ C Age	Marine ¹⁴ C Age	ΔR	Terrestrial ¹⁴ C Age	Marine ¹⁴ C Age	ΔR
5933	6568	261	6133	6568	33
5933	6477	169	6133	6477	-58
5933	6282	-26	6133	6282	-253
5933	6415	107	6133	6415	-120
6034	6568	158	6052	6568	141
6034	6477	67	6052	6477	50
6034	6282	-128	6052	6282	-145
6034	6415	5	6052	6415	-12

Table 1. Possible ΔR values for 4 terrestrial and 4 marine ¹⁴C ages where each group of 4 measurements passes a χ^2 test. ΔR range = -253 to +261 years; Average ΔR = 16 years; Standard deviation = 136 years; Standard error = 34 years.

Curve Employed	Method of Calculation	Calibrated Age Range and Associated Probability at 95.4%
IntCal 13	Subtract 400 years	cal AD 25 – 130
Intcal 13	Subtract 405 ± 40 years (Harkness, 1983)	20 cal BC – cal AD 225
Marine 13	Use $\Delta R = 0 \pm 0$	60 cal BC – cal AD 95
Marine 13	Use $\Delta R = -33 \pm 93$ (Reimer et al., 2002)	310 cal BC – cal AD 200
Marine 13	Use $\Delta R = -47 \pm 52$ (Russell et al., in preparation)	190 cal BC – cal AD100

Table 2. Changes in calibrated age range for a radiocarbon age of 2320 ± 20 y BP, with varying methods of calculation

Species	Number of individuals	Average $\delta^{13}\text{C}$ value (‰) in protein fraction
Periwinkle (<i>Littorina littorea</i>)	25	-15
Limpet (<i>Patella vulgata</i>)	45	-16
Cockle (<i>Cerastoderma edule</i>)	11	-16
Mussel (<i>Mytilus edulis</i>)	17	-16
Oyster (<i>Ostrea edulis</i>)	3	-20
Terrestrial animal protein	General value	-24
Marine protein	General Value	-15
C ₃ Plants	General value	-25

Table 3. Indicative $\delta^{13}\text{C}$ values for the protein fraction in a range of human dietary components. All mollusc samples were collected from the NW Scottish coast or the Orkney Isles. General values are from Hedges (2004).

Sample	^{14}C Age (y BP)	$\delta^{13}\text{C}$ (‰)	$\delta^{15}\text{N}$ (‰)	C/N ratio
SUERC-42896	434 ± 18	-18.7	+14.6	3.2
SUERC-42902	440 ± 17	-18.6	+15.0	3.2
OxA-27182	478 ± 25	-18.37	+15.0	n/q
OxA-27183	480 ± 25	-18.38	+15.3	n/q

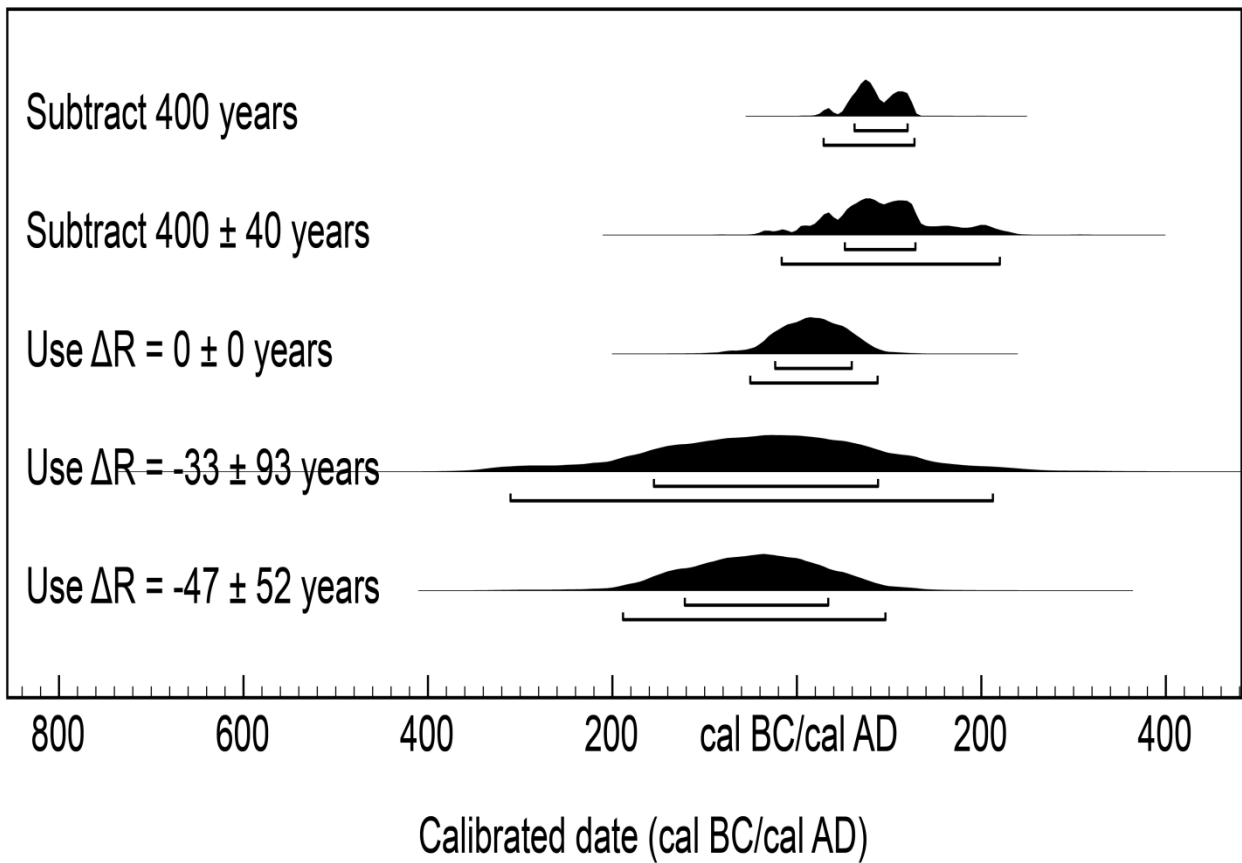
n/q: not quoted in report

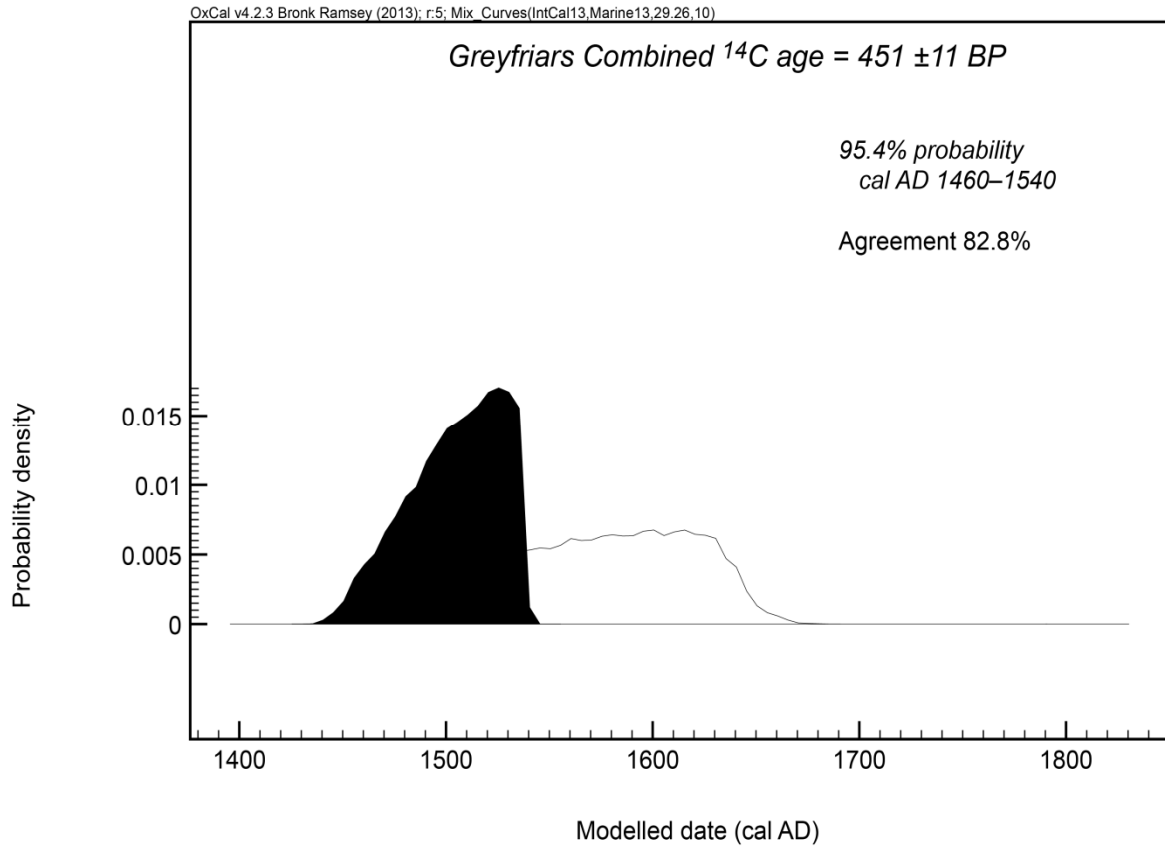
Table 4. ^{14}C and stable isotope data for rib samples purported to be Richard III.

Skeleton ID	Lab ID	¹⁴ C Age (y BP)	Calibrated Date (95.4% probability)	δ ¹³ C (‰)	δ ¹⁵ N (‰)	δ ³⁴ S (‰)	Diet	% Marine Diet ± 10%	Re-calibrated date (95.4% probability)
SK013	SUERC-41975	1123 ± 24	cal AD 820–990	−20.1	7.4	11.6	T	8	cal AD 770–1040
SK047	SUERC-41982	1005 ± 24	cal AD 990–1140	−20.2	8.5	9.8	T	7	cal AD 970–1190
SK053	SUERC-39955	1130 ± 30	cal AD 780–990	−20.1	7.7	10.3	T	8	cal AD 770–1030
SK007	SUERC-43994	1212 ± 29	cal AD 700–890	−19.2	10.6	12.3	M	20	cal AD 770–1020
SK056	SUERC-44122	1184 ± 29	cal AD 730–950	−19.3	10.9	14.9	M	19	cal AD 770–1040
SK009	SUERC-39947	1060 ± 30	cal AD 900–1030	−18.7	12.3	13.8	M	27	cal AD 1010–1230
SK016	SUERC-39952	2030 ± 30	150 cal BC– cal AD 50	−17.3	10.3	5.5	F	46	*cal AD 70–340
SK061	SUERC-39956	1560 ± 30	cal AD 420–570	−19.0	10.6	6.3	F	23	*cal AD 430–680
SK066	SUERC-39957	1705 ± 30	cal AD 250–410	−18.0	11.2	6.6	F	36	*cal AD 400–630

Table 5: Radiocarbon ages, stable isotope results, % marine diets, calibrated and recalibrated ages for Hofstaðir human bone collagen.

Calibrated date ranges are based on measured ¹⁴C ages following calibration with the IntCal 13 atmospheric calibration curve and OxCal v.4.2.4. Re-calibrated date ranges are based on mixed IntCal 13 and Marine 13 as described in the text, with the assumption that the δ¹³C value reflects the degree of marine resources in the diet. T = primarily a terrestrial diet, M = significant marine dietary component present, F = significant freshwater dietary component present. *Although adjusted dates are calibrated based on % of marine protein in the diet, these individuals favoured consumption of freshwater protein. As such, the adjusted calibrated date provides a *terminus post quem* for these individuals since there is no known method for correcting freshwater offsets due to the variability of the freshwater reservoir effect.





ACCEPTED MANUSCRIPT