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## Radiocarbon reservoir effects in human bone collagen from northern Iceland

Philippa L. Ascough <sup>a,\*</sup>, Mike J. Church <sup>b</sup>, Gordon T. Cook <sup>a</sup>, Elaine Dunbar <sup>a</sup>, Hildur Gestsdóttir <sup>c</sup>, Thomas H. McGovern <sup>d</sup>, Andrew J. Dugmore <sup>e</sup>, Adolf Friðriksson <sup>c</sup>, Kevin J. Edwards <sup>f,g,h</sup>

- <sup>a</sup> SUERC, Scottish Enterprise Technology Park, Rankine Avenue, East Kilbride G75 0QF, UK
- <sup>b</sup> Department of Archaeology, Durham University, South Road, Durham DH1 3LE, UK
- <sup>c</sup> Fornleifastofnun Íslands (Institute of Archaeology), Bárugata 3, 101 Reykjavík, Iceland
- <sup>d</sup> Hunter Bioarchaeology Laboratory, Hunter College CUNY, NYC 10021, USA
- e Institute of Geography, School of GeoSciences, University of Edinburgh, Drummond Street, Edinburgh EH9 8XP, UK
- f Department of Geography & Environment, University of Aberdeen, Elphinstone Road, Aberdeen AB24 3UF, UK
- <sup>g</sup> Department of Archaeology, University of Aberdeen, Elphinstone Road, Aberdeen AB24 3UF, UK
- h Clare Hall and McDonald Institute for Archaeological Research, University of Cambridge, UK

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#### ABSTRACT

Human bone collagen from a series of Icelandic human pagan graves was radiocarbon (14C) dated to aid understanding of early settlement (landnám) chronologies in northern Iceland. These individuals potentially consumed marine protein. The <sup>14</sup>C age of samples containing marine carbon requires a correction for the marine <sup>14</sup>C reservoir effect. The proportion of non-terrestrial sample carbon was quantified via measurement of carbon stable isotopes ( $\delta^{13}$ C) using a simple mixing model, based on  $\delta^{13}$ C measurements of archaeofaunal samples. Non-terrestrial carbon was also quantified in six pig bones from the archaeofaunal dataset. Assuming all non-terrestrial carbon in human and pig bone collagen was marine-derived, calibrated age ranges calculated using a mixed IntCal09/Marine09 calibration curve were consistent with an early settlement date close to landnám, but several samples returned prelandnám age ranges. Measurements of nitrogen stable isotopes ( $\delta^{15}N$ ) strongly suggest that many of the human bone collagen samples contain freshwater diet-derived carbon. Icelandic freshwater systems frequently display large freshwater <sup>14</sup>C reservoir effects, of the order of 10,000 <sup>14</sup>C years, and we suggest that the presence of freshwater carbon is responsible for the anomalously early ages within our dataset. In pig samples, the majority of non-terrestrial carbon is freshwater in origin, but in human samples the proportion of freshwater carbon is within the error of the marine component (+10%). This presents a major obstacle to assessing temporal patterns in the ages of human remains from sampled graves, although the majority of grave ages are within the same, broad, calibrated range.

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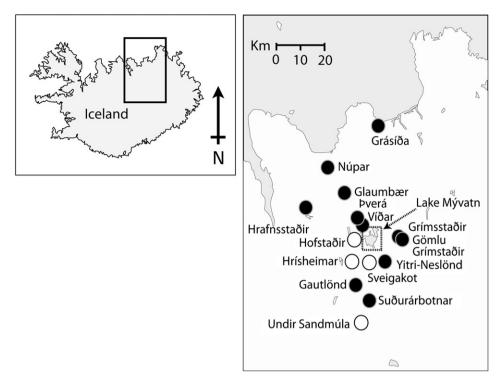
### 1. Introduction

The pristine landscape of Iceland was colonised from AD 871  $\pm$  2 (Grönvold et al., 1995) as part of the Viking (early Norse) landnám across the North Atlantic (Dugmore et al., 2005). Post- landnám Icelandic landscapes experienced large-scale human environmental impacts, climatic variation and societal changes (Vésteinsson, 1998, 2000; Buckland, 2000; Andrews et al., 2001; Dugmore et al., 2007; Lawson et al., 2007), yet a lack of detailed contemporary historical records means archaeological and palae-oenvironmental data are crucial for studying this initial settlement period. A key question is verifying the rapid timing of inland

settlement; midden deposits from various excavated settlements are in direct contact with the *landnám* tephra at Mývatnssveit (i.e. the region surrounding Lake Mývatn; Fig. 1), c. 60 km from the coast (McGovern et al., 2006a, 2007). This is parallelled by a considerable number of pagan graves running from the north Icelandic coast to the interior highlands (Fig. 1; Gestsdóttir, 1998; Eldjárn, 2000; Roberts, 2008). These pagan graves are likely to contain early inhabitants of Iceland, pre-dating the Christian conversion around AD 1000. To establish if these interments represented a single age range, or spatially variable ages dependent on the distance from the coast, bone collagen from human and animal bone from the graves was radiocarbon (<sup>14</sup>C) dated as part of the 'Landscapes circum*landnám*' project (Edwards et al., 2004; Dugmore et al., 2005).

A major consideration when <sup>14</sup>C dating human bone is whether any sample carbon (C) originated from a non-terrestrial reservoir. Terrestrial carbon sources include protein from domesticated land

<sup>\*</sup> Corresponding author. Tel.: +44 1355 223332; fax: +44 1355 229898. E-mail address: philippa.ascough@gla.ac.uk (P.L. Ascough).



**Fig. 1.** Locations of sites from which material was obtained for stable isotope ( $\delta^{13}$ C and  $\delta^{15}$ N) and radiocarbon ( $^{14}$ C) measurement. Left hand image indicates the study area within Iceland. Pagan grave sites are indicated by black circles, archaeofaunal sampling sites are indicated by white circles.

mammals (e.g. cattle), while non-terrestrial carbon sources include marine and freshwater fish and birds, and marine mammals (e.g. seals). The <sup>14</sup>C age of samples from the atmospheric and terrestrial biospheric carbon reservoirs are calibrated to a calendar year age span with the IntCalO9 atmospheric curve (Reimer et al., 2009), but the <sup>14</sup>C age of samples from other C reservoirs can be offset from that of contemporaneous atmospheric/terrestrial samples. This offset is known as a 'reservoir effect' and must be corrected for in order to produce accurate calibrated age ranges. The marine <sup>14</sup>C reservoir effect (MRE) results from radioactive decay of <sup>14</sup>C atoms during deep ocean water circulation (Stuiver and Braziunas, 1993; Ascough et al., 2005). In 100% marine samples, the MRE is quantified by calibration with the separate Marine09 curve, plus an additional local offset from the global average MRE, known as  $\Delta R$ (Stuiver and Braziunas, 1993; Ascough et al., 2005; Reimer et al., 2009). The <sup>14</sup>C age of bone collagen is a time-averaged integration of  $^{14}$ C in dietary protein consumed over  $\sim 10-30$  years prior to death (Ambrose and Norr, 1993; Hedges et al., 2007), meaning 14C ages from individuals that consumed large quantities of marine protein appear older than those of contemporaneous individuals that consumed 100% terrestrial diets (cf. Tauber, 1983; Yoneda et al., 2002; Bayliss et al., 2004). The importance of marine resources to Norse communities, even when located many kilometres inland (Einarsson, 1994; McGovern et al., 2006a), means that <sup>14</sup>C dating in the Viking Age North Atlantic can be problematic (e.g. Arneborg et al., 1999; Barrett et al., 2000; Ascough et al., 2006; Sveinbjörnsdóttir et al., 2010). Samples in this study were therefore assessed to identify <sup>14</sup>C measurements affected by the MRE and correction applied to the ages where possible.

 $^{14}\text{C}$  ages of bone collagen containing both terrestrial and marine C can be calibrated with a mixed IntCal09 and Marine09 calibration curve (Bronk Ramsey, 1998). The amount of marine carbon in the sample must be quantified, usually via its  $^{13}\text{C}/^{12}\text{C}$  stable isotope ratio ( $\delta^{13}\text{C}$  value) (Coplen, 1995). Bone collagen  $\delta^{13}\text{C}$  values predominantly reflect the  $\delta^{13}\text{C}$  of dietary protein; this is

significantly different for marine and terrestrial protein, where the  $\delta^{13} C$  of terrestrial herbivore tissue is typically c. -23 to -20% (e.g. DeNiro and Epstein, 1978), compared to c. -15 to -17% for marine fish (e.g. Ambrose and Norr, 1993; Jim et al., 2004; DeNiro and Epstein, 1978; Hobson, 1990). The proportion of marine C in bone collagen of terrestrial omnivores can be assessed on a mass balance basis:

$$\delta_{M} = f_{Terr} imes \delta_{Terr} + f_{Mar} imes \delta_{Mar}$$

Where:  $\delta_M$  = isotopic value of the mixture in the sample;  $f_{Terr}$ ,  $f_{Mar}$  = fraction of terrestrial and marine C, respectively (where  $f_{Terr}$ ,  $f_{Mar}$  = 1);  $\delta_{Terr}$ ,  $\delta_{Mar}$  = isotope values of terrestrial and marine C, respectively.

The simplest approach to calculate  $f_{Mar}$  is via a linear mixing model, as previously used to successfully calibrate <sup>14</sup>C ages of human bone collagen, including Viking period samples from the North Atlantic (cf. Arneborg et al., 1999; Sveinbjörnsdóttir et al., 2010). This approach requires  $\delta^{13}C$  end-member values for the bone collagen of a consumer existing on i) 100% terrestrial protein, and ii) 100% marine protein. These can be obtained from individuals known to have existed on the diets in question, or from measurements of dietary resources that are corrected for the diet-consumer trophic level fractionation. In either case, the accuracy of the calculated marine C proportions depends upon the selected endmember values (Dewar and Pfeiffer, 2010), which must be obtained from the same geographical region as the samples themselves (Hobson, 1999). This is because plant  $\delta^{13}$ C values, and hence herbivore tissue  $\delta^{13}$ C values, show wide geographical variation (McCarroll and Loader, 2004). In this study we measured the  $\delta^{13}$ C in geographically and temporally relevant samples of major terrestrial and marine protein sources. For a single species population accessing the same food resources, uncertainty in stable isotope-based dietary reconstructions can result from the range in isotopic values. This appears to be a consequence of individual

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