# Application of $^{34}$S analysis for elucidating terrestrial, marine and freshwater ecosystems: Evidence of animal movement/husbandry practices in an early Viking community around Lake Mývatn, Iceland

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## Abstract

Carbon and nitrogen stable isotope ratios ($\delta^{13}$C and $\delta^{15}$N) have been used widely in archaeology to investigate palaeodiet. Sulphur stable isotope ratios ($\delta^{34}$S) have shown great promise in this regard but the potential of this technique within archaeological science has yet to be fully explored. Here we report $\delta^{34}$S, $\delta^{13}$C and $\delta^{15}$N values for 129 samples of animal bone collagen from Skútustaðir, an early Viking age (*landnám*) settlement in north-east Iceland. This dataset represents the most comprehensive study to date of its kind on archaeological material and the results show a clear offset in $\delta^{34}$S values between animals deriving their dietary resources from terrestrial (mean = +5.6 ± 2.8‰), freshwater (mean = −2.7 ± 1.4‰) or marine (mean = +15.9 ± 1.5‰) reservoirs (with the three food groups being significantly different at $2\sigma$). This offset allows reconstruction of the dietary history of domesticated herbivores and demonstrates differences in husbandry practices and animal movement/trade, which would be otherwise impossible using only $\delta^{13}$C and $\delta^{15}$N values. For example, several terrestrial herbivores displayed enriched bone collagen $\delta^{34}$S values compared to the geology of the Lake Mývatn region, indicating they may have been affected by sea-spray whilst being pastured closer to the coast, before being traded inland. Additionally, the combination of heavy $\delta^{15}$N values coupled with light $\delta^{34}$S values within pig bone collagen suggests that these omnivores were consuming freshwater fish as a significant portion of their diet. Arctic foxes were also found to be consuming large quantities of freshwater resources and radiocarbon dating of both the pigs and foxes confirmed previous studies showing that a large freshwater radiocarbon ($^{14}$C) reservoir effect exists within the lake. Overall, these stable isotope and $^{14}$C data have important implications for obtaining a fuller reconstruction of the diets of the early Viking settlers in Iceland, and may allow a clearer identification of the marine and/or freshwater $^{14}$C reservoir effects that are known to exist in human bone collagen.
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## 1. INTRODUCTION

Extensive controlled feeding studies of modern terrestrial, freshwater and marine species have shown that that diet can directly influence carbon ($\delta^{13}C$) and nitrogen ($\delta^{15}N$) stable isotope values in the tissues of the consumer (DeNiro and Epstein, 1978, 1981). Archaeologists were very quick to exploit this theory and stable isotope analysis of preserved bone collagen is now routinely utilised in palaeodietary reconstruction studies, providing archaeologists with a detailed insight into prehistoric diet (Van der Merwe and Vogel, 1978; Tauber, 1981; Schoeninger et al., 1983).

Recent advances in continuous-flow isotope-ratio mass spectrometry (CF-IRMS) have allowed sulphur isotopes ($\delta^{34}S$) to be measured from organic and inorganic materials (Giesemann et al., 1994; Fry et al., 1996; Grassineau et al., 2001; Yun et al., 2005; Fritzsche and Tichomirowa, 2006; Grassineau, 2006; Fry, 2007; Hansen et al., 2009) and over the past decade there has been a marked increase in the use of sulphur isotopes in conjunction with carbon and nitrogen isotopes to aid our understanding of the diet and movement of prehistoric animals and humans (Macko et al., 1999; Richards et al., 2001, 2003; Hu et al., 2009; Vika, 2009; Craig et al., 2010; Howcroft et al., 2012; Nehlich et al., 2012; Oelze et al., 2012a,b). Sulphur isotopes have also been exploited to explore the variability in terrestrial-, marine- and freshwater-based diets (Craig et al., 2006; Privat et al., 2007; Nehlich et al., 2010, 2011; Lamb et al., 2012), have been used as indicators of environmental changes (Wadleigh, 2003; Newton and Bottrell, 2007; Yun et al., 2010) and are regularly utilised in food authentication studies (Bahar et al., 2008; Rummel et al., 2010; Tanz and Schmidt, 2010; Osorio et al., 2011).

Previous findings from the Lake Mývatn region of north-east Iceland have demonstrated a significant overlap between the $\delta^{15}N$ values of both modern and archaeological-age terrestrial herbivore bone collagen and the $\delta^{15}N$ values of modern and archaeological-age freshwater fish. $\delta^{13}C$ values of freshwater biota were also found to be similar to those of marine resources (Ascough et al., 2010), and consequently, separation of herbivores, freshwater fish and marine fish as components of human diet is difficult using only $\delta^{13}C$ and $\delta^{15}N$ analyses. Likewise, complications were encountered when human bone collagen samples from the same region were radiocarbon ($^{14}C$) dated, as the exact proportion of freshwater and marine carbon consumed was indeterminable, which prevented ages being corrected for both marine and freshwater $^{14}C$ reservoir effects (Ascough et al., 2012). Freshwater and marine radiocarbon reservoir effects (FRE and MRE, respectively) are $^{14}C$ age offsets between $CO_2$ in the atmosphere and the freshwater or marine carbon reservoirs. The MRE arises because of the extended residence time of carbon in the global marine reservoir, during which time radioactive decay of the $^{14}C$ occurs. It has a global average value of approximately 400 $^{14}C$ years for surface waters and can be corrected for in order to produce a truer calendar age range (Reimer et al., 2009). FREs are brought about by the input of low $^{14}C$-activity carbon (e.g. carbon from dissolution of geological carbonates or from high temperature geothermal water–rock interac-

tions), or restriction of atmosphere-water $CO_2$ exchange (e.g. via density stratification or ice cover). Unlike the MRE, a universal amendment cannot be implemented as the magnitude of FREs is site dependent and can fluctuate significantly (Ascough et al., 2011; Keaveney and Reimer, 2012).

The purpose of this study was to utilise $\delta^{34}S$ stable isotope measurements in animal and bird bone collagen from remains found in midden deposits at a Viking farmstead on Lake Mývatn in an attempt to distinguish between terrestrial, marine and freshwater dietary components. In turn, this could allow the identification of terrestrial, freshwater and marine components in the human diet. To date, this is the first study of its kind to examine sulphur isotopes in bone collagen from archaeological remains found in Iceland. It is also the largest, single-site sulphur isotope study to be undertaken, in which 129 bones of domesticated and wild fauna from Skútustaðir were analysed. These data were produced, together with C and N stable isotope ratio measurements and $^{14}C$ age measurements, as part of a multi-isotope approach, building on previous work to reconstruct animal diet and human activity in the region (Ascough et al., 2007, 2010, 2012).

## 2. BACKGROUND

### 2.1. Geographical and historical information

The animal bones analysed in this study were from Skútustaðir, an archaeological site to the south of Lake Mývatn (meaning "the lake of midges" in Icelandic) in the north-eastern highlands of Iceland. Famed, as its name suggests, for its abundant insect life, this shallow lake, located ∼50 km inland and at an altitude of 277 m above sea level (Fig. 1), is a sanctuary for breeding waterfowl (Einarsson, 2004; Gardarsson, 2006). The region has been documented as an area of major archaeological importance with respect to the settlement of Viking communities during the *landnám* from around AD 870 onwards (Vésteinsson, 1998; McGovern et al., 2007; Einarsson and Aldred, 2011). Radiocarbon dating of terrestrial animal remains and tephrochronological studies from various sites surrounding Lake Mývatn have shown that settlers populated the region from the late 9th century (McGovern et al., 2006, 2007). The presence of these inhabitants is thought to have had a large environmental impact on the area, with the introduction of grazing livestock and rapid deforestation (Hallsdóttir, 1987), leading to significant soil erosion (Arnalds et al., 1997; Vésteinsson et al., 2002; Dugmore et al., 2005; Lawson et al., 2007).

### 2.2. Geology of the Lake Mývatn area

The area surrounding Lake Mývatn is volcanic in nature, with igneous rocks of the tholeiitic series dominating the landscape. The series is split into three subsections: (1) basaltic rocks, which are most abundant, comprising of picrite, olivine tholeiite and tholeiite; (2) intermediate rocks which include icelandite and basaltic icelandite; and (3) silicic rocks which include dacite and rhyolite (Jakobs-
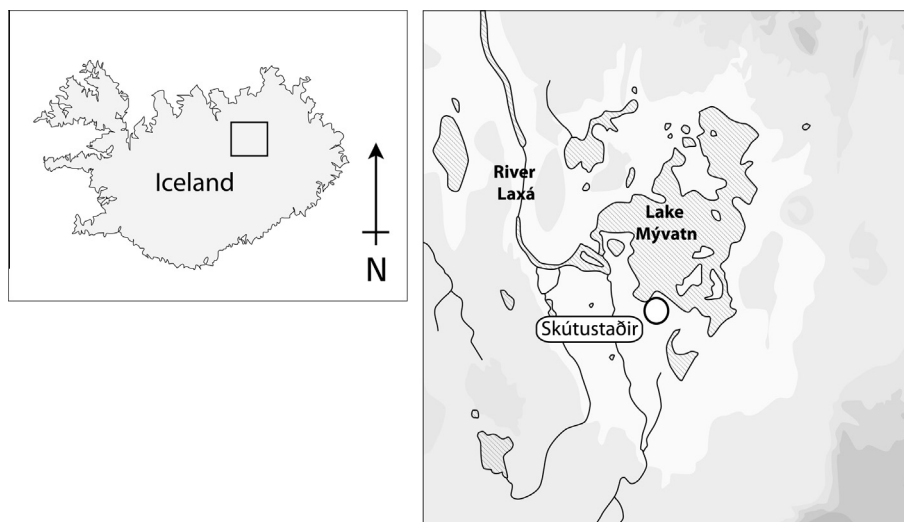
Fig. 1. Location of Skútustaðir from which material was obtained for stable isotope ($\delta^{13}C$, $\delta^{15}N$ and $\delta^{34}S$) and radiocarbon ($^{14}C$) measurements.

son et al., 2008). Porous lava fields dominate the area, leaving the surface characteristically devoid of water. The lake has two major basins, Ytriflói (north basin), which is fed by hot springs from the Námafjall geothermal field, and Syðriflói (south basin), which is fed by cold springs along its eastern shores (Kristmannsdóttir and Ármannsson, 2004). As groundwater springs supply most of Lake Mývatn's water and there is an absence of surface water in the area for it to mix with, the chemical makeup of the water entering the lake is very stable. Before draining into the River Laxá in the west, the geothermal waters provide Lake Mývatn with plentiful supplies of silica and sulphate, whilst cooler waters deliver phosphate to the lake (Kristmannsdóttir and Ármannsson, 2004).

### 2.3. Isotope geochemistry of Iceland

The lithosphere and hydrosphere store the majority of the earth's sulphur supplies, with sulphides in shale and sulphates in evaporites exhibiting $\delta^{34}S$ values between $-40‰$ and $+30‰$ (Claypool et al., 1980; Strauss, 1997) and sulphate in marine water providing a very isotopically uniform reservoir of $\delta^{34}S = +21‰$ (Rees et al., 1978). In coastal regions, sulphur-containing particles can be propelled inland in a process known as the sea-spray effect, causing soil $\delta^{34}S$ values to be similar to that of seawater (Wadleigh et al., 1994).

Intensive weathering of igneous rocks causes leached sulphides to filter into ground and stream water systems, enabling plants to accumulate the oxidised sulphate form in their roots. Oxidation of pyrite ($FeS_2$) produces negligible isotopic fractionation (Nakai and Jensen, 1964; Taylor and Wheeler, 1984), whilst plant $\delta^{34}S$ values are on average 1.5‰ depleted compared to their sulphate source (Trust and Fry, 1992). Analysis of total sulphur in volcanic rocks from the Krafla-Námafjall fissure swarm neighbouring Lake Mývatn provided $\delta^{34}S$ values that ranged between $-2.0‰$ and $+4.2‰$ (mean: $-0.8‰$) (Torssander, 1989),

whilst examination of transitional basaltic and rhyolitic rocks from the Katla Volcanic Centre in southern Iceland gave similar $\delta^{34}S$ values, ranging between $-1.8‰$ and $+2.4‰$ (Hildebrand and Torssander, 1998). As isotopic fractionation between plants and sulphates deposited in the soil from the weathering of local bedrock is relatively small, $\delta^{34}S$ values of Lake Mývatn flora should be similar to those reported by Torssander (1989). However, Icelandic lava fields are sparsely vegetated with lichen and moss (Bjarnason, 1991), which can accumulate their sulphur directly from atmospheric sulphur dioxide ($SO_2$) with very little isotopic fractionation (Krouse, 1977). $SO_2$ gas produced during the eruption of Krafla in July 1980 yielded $\delta^{34}S$ values between $-1.8‰$ and $+3.4‰$ (Torssander, 1988), however, it is conceivable that atmospheric $SO_2$ in Iceland has varied with time due to the volcanic nature of the island. Wet deposition of sulphate from the aqueous oxidation of atmospheric $SO_2$ can provide soil with an additional source of sulphur and isotopic fractionation during this process can be very large (ca. $-11‰$ to $+17‰$) (Harris et al., 2012). However, the amount of sulphate deposited has been shown to be negligible in areas where $SO_2$ emissions are high (Nriagu and Coker, 1978), and compared to other parts of Iceland, the Mývatn region has little rain- or snowfall (Einarsson, 1979).

The eruption of the Grímsvötn volcano in 1996, which is situated below the Vatnajökull Glacier in south-east Iceland, caused the Skeiðará River to flood, and water samples taken before and during the early stages of the overflow revealed a post-eruption increase in sulphur concentration. Analysis of sulphate in the floodwaters demonstrated that $\delta^{34}S$ values varied between $+7.9‰$ and $+9.0‰$, suggesting that sulphurous magmatic gases were oxidised to sulphate upon dissolution in water (Gíslason et al., 2002). Together with water samples taken from kettle-hole lakes that had formed since the 1996 glacier-outburst flood, further groundwater, geothermal spring water and Skeiðará River samples were retrieved between 1998 and 2001 from the

Skeiðarársandur outwash plain (Robinson et al., 2009). $\delta^{34}S$ values from river sulphates ranged from +3.4‰ to +8.8‰, with the higher value again attributed to magmatic sulphate from the Grímsvötn caldera, whilst the lower end value was measured during normal discharge; both values fall within the range of +2‰ to +10‰ reported by Gíslason and Torssander (2006). Groundwater sulphate $\delta^{34}S$ values varied between −0.3‰ and +5.3‰, whilst sulphate in geothermal spring water had an average $\delta^{34}S$ value of +4.1‰. Water from the kettle-hole lakes was found to be depleted in $^{34}S$, with $\delta^{34}S$ values ranging between −1.7‰ and 0.0‰, which could be attributed to dissolved sulphate originating from igneous sulphide minerals. Samples taken from the Hekla cold springs in southern Iceland, fifteen years after the volcano last erupted, revealed that magmatic degassing into groundwater was still occurring. Dissolved sulphate in these water samples displayed $\delta^{34}S$ values between +1.5‰ and +4.3‰, with magmatic sulphate estimated to have had a $\delta^{34}S$ value of around +7.0‰ (Holm et al., 2010). Thus, the numerous volcanic eruptions that have occurred since Iceland was first settled are likely to have influenced the $\delta^{34}S$ values of all water sources feeding Lake Mývatn and its surrounding landscape. However, whilst Icelandic rock and water samples have demonstrated $\delta^{34}S$ values that span from −2‰ to +10‰, and atmospheric $SO_2$ $\delta^{34}S$ values in the Mývatn region may vary between −1.8‰ and +3.4‰, what is evident is that they are all isotopically very distinct from the $\delta^{34}S$ value of seawater (Fig. 2).

## 2.4. Isotope biochemistry of Iceland

$\delta^{13}C$ analysis of various plants and lichens from four lakes in northern Iceland provided values ranging between −30.9‰ and −23.3‰ (Wang and Wooller, 2006). Likewise, the analysis of moss, grass, willow and liverwort samples from the geothermal area of Kerlingarfjöll in central Iceland produced $\delta^{13}C$ values between −28.8‰ and −20.4‰ (Skrzypek et al., 2008). Ascough et al. (in press) measured $\delta^{13}C$ on a variety of modern flora from four sites close to Lake Mývatn and at one location approximately 5 km to the west of the lake, and found that the vegetation ranged from −31.6‰ to −26.9‰. These results are within the expected range for plants in the northern hemisphere following a $C_3$ photosynthetic pathway, with the more enriched value of −20.4‰ from Kerlingarfjöll being attributed to the moss growing in a colder climate (Skrzypek et al.,

2007). Aquatic plant samples from one site had $\delta^{13}C$ values averaging −13.3‰, which is characteristic of freshwater plants in Iceland (Wang and Wooller, 2006). Previous stable isotope studies of archaeological bone samples discovered at various sites surrounding Lake Mývatn indicate that $\delta^{13}C$ values for terrestrial animals ranged from −22.1‰ to −20.3‰, whilst modern and archaeological freshwater fish displayed $\delta^{13}C$ values from −16.0‰ to −7.9‰. Omnivorous pigs and various birds displayed a large range of $\delta^{13}C$ values (−22.5‰ to −16.9‰ and −24.8‰ to −7.9‰, respectively), reflecting the mixed terrestrial, freshwater and possibly marine diet they would have been consuming (Ascough et al., 2007, 2010, 2012, in press). Although cod and haddock bones have been recovered at Skútustaðir, prior to this study, no stable isotope analysis had been undertaken on these samples. However, cod from four archaeological sites in the north-east Atlantic yielded $\delta^{13}C$ values between −14.7‰ and −11.3‰ (Barrett et al., 2008, 2011; Russell et al., 2011).

It is well established that, although $^{13}C$ trophic level shifts are small, $^{15}N$ values have been shown to shift between +3‰ and +5‰ with each trophic level in marine and terrestrial food chains (Schoeninger and DeNiro, 1984). Given that terrestrial plants have $\delta^{15}N$ values ranging between approximately 0‰ and +5‰, herbivores and carnivores should accordingly exhibit $\delta^{15}N$ values of ~+4‰ to +9‰ and ~+8‰ to +13‰, respectively. Within the marine environment, $\delta^{15}N$ can range between ~+15‰ to +20‰ as a consequence of the food chains being considerably longer than in the terrestrial biosphere (DeNiro and Epstein, 1981; Schoeninger et al., 1983; Schoeninger and DeNiro, 1984). However, organisms lower down in the marine food chain such as gastropods, molluscs, Polychaeta and Maxillopoda demonstrate lighter $\delta^{13}C$ (−23.2‰ to −17.1‰) and $\delta^{15}N$ values (+5.9‰ to +9.7‰) (Mateo et al., 2008). Freshwater fish have also displayed enriched $\delta^{15}N$ values; however studies have shown that some species have $\delta^{13}C$ and $\delta^{15}N$ values that are similar to those observed within a terrestrial environment (Dufour et al., 1999). Therefore, the introduction of a third stable isotope, namely sulphur, to aid in the distinction between these two food groups, is potentially highly advantageous.

Wang and Wooller (2006) carried out $\delta^{15}N$ analysis on the plants and lichens recovered from the four sites mentioned previously in northern Iceland and found that values ranged from −12.4‰ to +5.6‰, with many of the samples
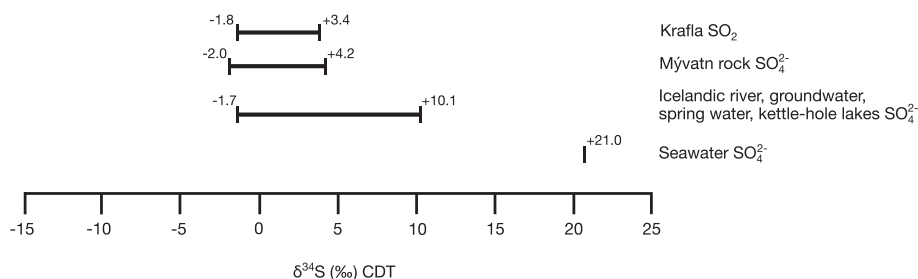


Fig. 2. $\delta^{34}S$ values for various Icelandic sulphur sources.

having values lower than −6.0‰. Similarly, the analysis of moss, grass, willow and liverwort specimens from Kerlingarfjöll produced $\delta^{15}N$ values between −5.5‰ and −1.7‰ (Skrzypek et al., 2008). Ascough et al. (in press) determined that modern flora from around Lake Mývatn had $\delta^{15}N$ values between −9.1‰ and +6.5‰. It has been noted that in soils depleted in phosphorous, plants and lichens that take up ammonia from the atmosphere are capable of generating negative $\delta^{15}N$ values (Erskine et al., 1998; McKee et al., 2002). Ascough et al. (2007, 2010, 2012, in press) found that $\delta^{15}N$ values for modern and archaeological terrestrial animals ranged between −1.5‰ and +5.9‰, whilst freshwater fish displayed $\delta^{15}N$ values from +3.1‰ to +8.5‰, and north-east Atlantic cod yielded $\delta^{15}N$ values from +11.9‰ to +15.4‰ (Barrett et al., 2008, 2011; Russell et al., 2011). Again, the $\delta^{15}N$ values for pigs and birds displayed a large range of values (−1.2‰ to +8.7‰ and −3.7‰ and +16.4‰, respectively), and are typical of a mixed diet. A comparison of the various $\delta^{13}C$ and $\delta^{15}N$ ranges for Icelandic terrestrial and aquatic plants and fauna from the Lake Mývatn region is illustrated in Fig. 3.

Oceanic primary producers demonstrate sulphate $\delta^{34}S$ values between +17‰ and +21‰ (Peterson and Fry, 1987), however, $\delta^{34}S$ values for organisms living within a freshwater environment have been shown to vary anywhere between −22‰ and +20‰ due to differing sulphur sources in the local geology as well as anaerobic bacteria residing within lakes and rivers, which can reduce sulphate ions to hydrogen sulphide ($H_2S$) (Faure, 1977; Peterson and Fry, 1987). For mammals to successfully thrive, sulphur-containing biochemical compounds, such as the essential amino acids, methionine and cysteine, need to be acquired from the diet. Studies have demonstrated that there is a negligible trophic level shift for sulphur isotopes compared to the large range of $\delta^{34}S$ values shown within terrestrial, freshwater and marine environments (Peterson et al., 1985; Richards et al., 2003). Therefore, consumers eating produce from within the Lake Mývatn vicinity should have similar $\delta^{34}S$ values to the surrounding terrestrial vegetation. Currently, there are no published $\delta^{34}S$ values for either animal or human archaeological remains from Iceland, and globally only a small number of sulphur isotope studies have been undertaken on archaeological material. How-

ever, this is now changing rapidly and more recently an increasing number of archaeological studies have utilised sulphur isotope analysis as part of their investigations (Privat et al., 2007; Vika, 2009; Nehlich et al., 2011). Nevertheless, many of these findings could be seen to be limited in that they are either lacking in sample numbers, or where a larger sample set has been analysed, they have originated from multiple sites (Richards et al., 2001; Hu et al., 2009; Nehlich et al., 2010).

# 3. METHODOLOGY

## 3.1. Sampling location and materials

Skútustaðir, situated on the southern shore of Lake Mývatn (65° 57′ N 17° 03′ W), was excavated during the NSF-funded IPY program "*Long Term Human Ecodynamics in the Norse North Atlantic: cases of sustainability, survival, and collapse*". After the discovery of an archaeological midden in 2007, the site was explored in greater detail during a series of excavations between 2008 and 2010 (Edwald and McGovern, 2009, 2010; Hicks et al., 2009; Hicks, 2010; Hicks and Pálsdóttir, 2011), and a large number of animal bones and artefacts were discovered. The midden site is on a natural rise in the landscape, and due to the porous nature of the bedrock, drainage conditions are good and no pockets of waterlogged sediments were noted. The pH of the soil is around 6.5, providing favourable conditions for preservation of animal bones. The work discussed in this paper deals solely with excavations carried out during the 2008 and 2009 field trips and a list of all samples analysed with their stratigraphic context and chronology is presented in the Supplementary data section.

## 3.2. Extraction of bone collagen

A modified version of the Longin method was used to extract the collagen component from the animal bones (Longin, 1971). Sample surfaces were initially cleaned using a Dremel® multi-tool, before they were lightly crushed into smaller fragments and immersed in 1 M HCl for approx. 24 h to effect demineralisation. The acid was then decanted and samples were rinsed with ultra-pure water to remove any remaining dissociated carbonates, acid soluble
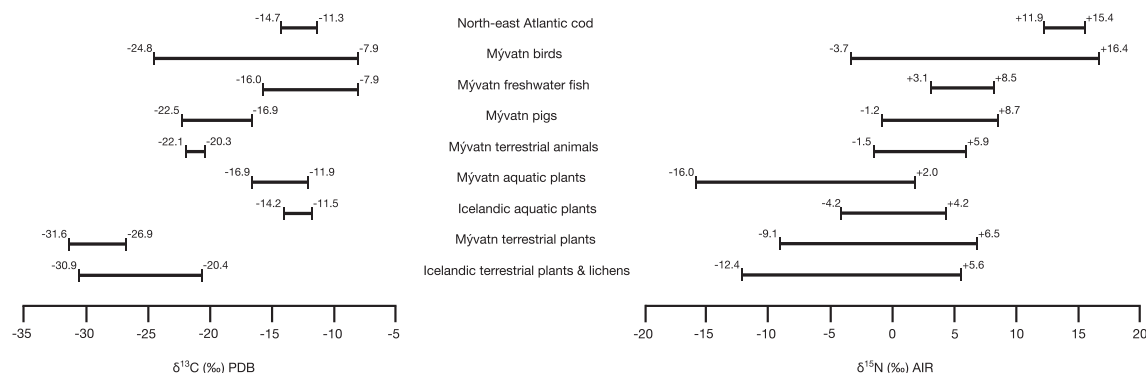


Fig. 3. $\delta^{13}C$ and $\delta^{15}N$ values for various flora and fauna from around Iceland and the Lake Mývatn region.

contaminants and solubilised bioapatite. The gelatinous-like material was heated gently to ~80 °C in ultra-pure water to denature and solubilise the collagen. After cooling, the solution was filtered, reduced to approx. 5 ml and freeze-dried.

### 3.3. Carbon, nitrogen and sulphur isotope ratio analyses

$\delta^{13}C$, $\delta^{15}N$ and $\delta^{34}S$ stable isotope measurements were obtained using a continuous-flow isotope ratio mass spectrometer (Thermo Scientific Delta V Advantage (Bremen, Germany)) coupled to a Costech ECS 4010 elemental analyser (EA) (Milan, Italy) fitted with a pneumatic autosampler. Samples were weighed into tin capsules (~600 μg for $\delta^{13}C$ and $\delta^{15}N$ and ~10 mg for $\delta^{34}S$) and the $\delta^{13}C$ and $\delta^{15}N$ values measured in one of two ways. The EA was coupled to the mass spectrometer via a ConfloIII™ and samples were combusted in a reactor containing chromium oxide and silvered cobaltous/cobaltic oxide at 1020 °C to produce $N_2$ and $CO_2$. The gases were then passed over a reduction reactor containing reduced copper wires at 650 °C. A magnesium perchlorate trap was used to eliminate water produced during the combustion process and the gases were separated in a 3 m stainless steel Porapak QS 50–80 mesh GC column heated to 45 °C. Alternatively, the EA was coupled via a ConfloIV™ and samples were combusted in a single reactor containing tungstic oxide and copper wires at 1020 °C to produce $N_2$ and $CO_2$. The gases were then separated in a 2 m stainless steel Porapak QS 50–80 mesh GC column heated to 70 °C. The latter system was used to obtain $\delta^{34}S$ values and the column was heated to 90 °C to separate $SO_2$. Helium (100 ml/min) was used as a carrier gas throughout the procedure. $N_2$, $CO_2$ and $SO_2$ entered the mass spectrometer via an open split arrangement within the ConfloIII™/ConfloIV™ and were analysed against their corresponding reference gases.

For every ten unknown samples, in-house gelatine standards, which are calibrated to the international reference materials USGS40 (−26.39‰), USGS41 (+37.63‰), IAEA-CH-6 (−10.45%), USGS25 (−30.41‰), IAEA-N-1 (+0.43‰) and IAEA-N-2 (+20.41‰), were run in duplicate. Results are reported as per mil (‰) relative to the internationally accepted standards VPDB and AIR with $1\sigma$ precisions of ±0.2‰ and ±0.3‰ for $\delta^{13}C$ and $\delta^{15}N$, respectively. All animals analysed had C:N atomic ratios that fell within the range of 2.9–3.6, indicating good bone collagen preservation (DeNiro, 1985). For $\delta^{34}S$ analysis, two internal standards, which are calibrated to the international reference materials IAEA-S-1 (−0.3‰), IAEA-S-3 (−32.55‰) and IAEA-S-4 (+16.90‰), were run for every five unknown samples. Results are reported as per mil (‰) relative to the internationally accepted standard VCDT and the precision was ±0.6‰. 25% of $\delta^{34}S$ analyses were carried out in duplicate; there were no significant differences in the reproducibility of the results.

Nehlich and Richards (2009) analysed a variety of mammalian and fish bone archaeological samples, with the objective of introducing quality control standards for measuring sulphur isotopes in bone collagen. They found that, on average, mammalian and bird bone collagen had an atomic C:S ratio of 600 ± 300, an atomic N:S ratio of 200 ± 100 and contained between 0.15% and 0.35% sulphur, whilst fish bone collagen was found to have an atomic C:S ratio of 175 ± 50, an atomic N:S ratio of 60 ± 20 and contained between 0.4% and 0.8% sulphur. A small number of individual samples in this study fell outside the above ranges and are excluded from the discussion (shown in italics and bold italics in their respective data tables in Supplementary data section). Two samples (highlighted by an asterisk in their respective data tables) have still been included as their values fall within the desired ranges when corrected for weighing errors on the analytical balance.

### 3.4. Radiocarbon dating

Radiocarbon ages were obtained from selected samples in this study. $CO_2$ was generated from collagen via combustion following the method of Vandeputte et al. (1996). Following cryogenic purification, $\delta^{13}C$ was measured on an aliquot of the $CO_2$ for normalization of sample $^{14}C/^{13}C$ ratios. This was achieved on a VG SIRA 10 isotope ratio mass spectrometer, using NBS 22 (oil) and NBS 19 (marble) as standards. The method of Slota et al. (1987) was used to convert a 3 ml aliquot of the $CO_2$ to graphite for $^{14}C$ measurement by accelerator mass spectrometry (AMS). Sample $^{14}C/^{13}C$ ratios were measured with carbon in the +1 charge state on the SUERC SSAMS at 245 keV. Calibrated age ranges at $2\sigma$ were obtained from sample $^{14}C$ ages using the atmospheric IntCal09 curve (Reimer et al., 2009) and OxCal version 4.1 (Bronk Ramsey, 1995, 2001).

### 4. RESULTS

A summary of the stable isotope results is presented in Table 1 and plotted in Fig. 4. All stable isotope and radiocarbon age measurements together with sampling areas and stratigraphic contexts analysed in this study, can be found in Tables S1–S9 in the Supplementary data section.

### 4.1. Terrestrial herbivores

Cows ($n = 32$): $\delta^{13}C$ values ranged from −22.5‰ to −20.6‰ (mean = −21.5 ± 0.4‰), $\delta^{15}N$ values from +1.1‰ to +5.6‰ (mean = +3.9 ± 1.0‰) and $\delta^{34}S$ values from −1.0‰ to +13.9‰ (mean = +4.1 ± 3.2‰), respectively.

Caprines (sheep and goats) ($n = 48$): $\delta^{13}C$ values ranged from −22.0‰ to −20.4‰ (mean = −21.2 ± 0.4‰), $\delta^{15}N$ values from −0.1‰ to +5.5‰ (mean = +2.5 ± 1.1‰) and $\delta^{34}S$ values from +2.3‰ to +12.3‰ (mean = +6.7 ± 1.9‰).

Horses ($n = 5$): $\delta^{13}C$ values ranged from −22.4‰ to −21.4‰ (mean = −21.8 ± 0.4‰), $\delta^{15}N$ values from +0.6‰ to +3.6‰ (mean = +1.9 ± 1.3‰), and $\delta^{34}S$ values from +1.4‰ to +10.2‰ (mean = + 5.7 ± 3.2‰).

### 4.2. Freshwater fish

The analysis of trout ($n = 5$) and char ($n = 7$) bones yielded $\delta^{13}C$ values that ranged from −9.8‰ to −9.3‰ (mean = −9.6 ± 0.2‰) and −11.4‰ to −9.1‰

Table 1
Mean and standard deviations ($1\sigma$) of bone collagen $\delta^{34}$S, $\delta^{13}$C and $\delta^{15}$N values for animals from Skútustaðir, Iceland.

| Species | N | $\delta^{34}$S [‰] | S wt% | $\delta^{13}$C [‰] | $\delta^{15}$N [‰] |
|---|---|---|---|---|---|
| Cow | 32 | $4.1 \pm 3.2$ | $0.21 \pm 0.05$ | $-21.5 \pm 0.4$ | $3.9 \pm 1.0$ |
| Sheep/goat | 48 | $6.7 \pm 1.9$ | $0.21 \pm 0.05$ | $-21.2 \pm 0.4$ | $2.5 \pm 1.1$ |
| Horse | 5 | $5.7 \pm 3.2$ | $0.20 \pm 0.02$ | $-21.8 \pm 0.4$ | $1.9 \pm 1.3$ |
| Trout | 5 | $-2.4 \pm 1.5$ | $0.53 \pm 0.04$ | $-9.6 \pm 0.2$ | $6.1 \pm 0.7$ |
| Char | 7 | $-3.0 \pm 1.3$ | $0.58 \pm 0.03$ | $-10.0 \pm 0.8$ | $5.9 \pm 0.5$ |
| Haddock | 3 | $14.0 \pm 1.8$ | $0.46 \pm 0.03$ | $-14.3 \pm 0.3$ | $12.6 \pm 0.3$ |
| Cod | 6 | $16.8 \pm 0.9$ | $0.50 \pm 0.02$ | $-14.2 \pm 0.4$ | $13.9 \pm 0.5$ |
| Seal | 6 | $15.9 \pm 1.0$ | $0.21 \pm 0.05$ | $-15.3 \pm 0.5$ | $12.7 \pm 0.5$ |
| Pig | 3 | $5.3 \pm 2.7$ | $0.17 \pm 0.01$ | $-19.5 \pm 1.0$ | $8.5 \pm 1.7$ |
| Birds | 11 | $3.0 \pm 5.0$ | $0.29 \pm 0.03$ | $-13.6 \pm 4.2$ | $6.5 \pm 3.9$ |
| Arctic fox | 3 | $1.4 \pm 0.7$ | $0.25 \pm 0.03$ | $-14.9 \pm 1.3$ | $9.0 \pm 1.5$ |
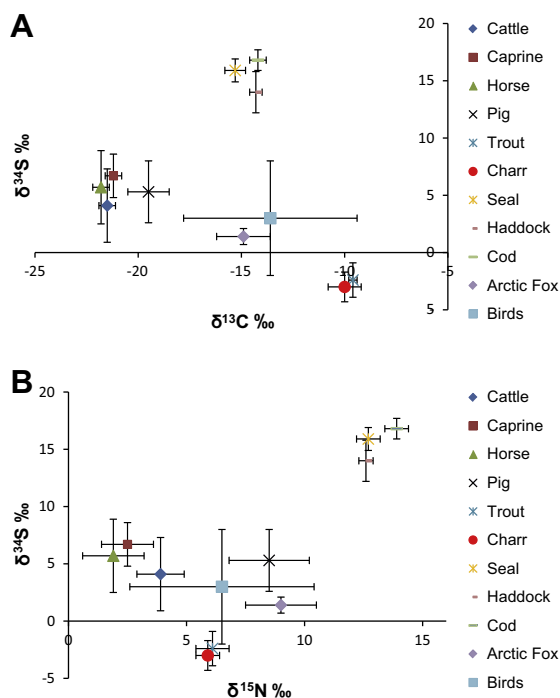


Fig. 4. Mean $\delta^{13}$C vs. $\delta^{34}$S (A) and $\delta^{15}$N vs. $\delta^{34}$S (B) for Skútustaðir animal bone collagen samples. Error bars show standard deviations ($1\sigma$) from the mean.

(mean $= -10.0 \pm 0.8‰$), respectively. $\delta^{15}$N values ranged from $+5.0‰$ to $+6.8‰$ (mean $= +6.1 \pm 0.7‰$) and $+5.2‰$ to $+6.8‰$ (mean $= +5.9 \pm 0.5‰$), respectively, whilst $\delta^{34}$S values ranged from $-4.2‰$ to $-0.2‰$ (mean $= -2.4 \pm 1.5‰$) and $-4.3‰$ to $-0.4‰$ (mean $= -3.0 \pm 1.3‰$), respectively.

### 4.3. Marine fish

Haddock ($n = 3$) and cod bones ($n = 6$) provided $\delta^{13}$C values from $-14.6‰$ to $-14.0‰$ (mean $= -14.3 \pm 0.3‰$) and $-14.7‰$ to $-13.5‰$ (mean $= -14.2 \pm 0.4‰$), respectively. $\delta^{15}$N values ranged from $+12.2‰$ to $+12.8‰$ (mean $= +12.6 \pm 0.3‰$), and $+13.3‰$ to $+14.5‰$ (mean $= +13.9 \pm 0.5‰$), respectively, whilst $\delta^{34}$S values

varied from $+12.4‰$ to $+15.9‰$ (mean $= +14.0 \pm 1.8‰$), and $+15.6‰$ and $+17.5‰$ (mean $= +16.8 \pm 0.9‰$), respectively. Haddock bones were on average $1.3‰$ less enriched in nitrogen and $2.8‰$ less enriched in sulphur than cod bones. Although both fish are carnivores, adult cod are slightly higher in the marine food web than haddock, and they have been known to eat smaller cod, hence the difference in $\delta^{15}$N and $\delta^{34}$S values.

### 4.4. Marine mammals

Seal bones ($n = 6$) $\delta^{13}$C values ranged from $-16.3‰$ to $-14.8‰$ (mean $= -15.3 \pm 0.5‰$), $\delta^{15}$N values from $+12.1‰$ to $+13.3‰$ (mean $= +12.7 \pm 0.5‰$) and $\delta^{34}$S values from $+14.3‰$ to $+16.8‰$ (mean $= +15.9 \pm 1.0‰$).

### 4.5. Omnivorous mammals

Pig bone ($n = 3$) $\delta^{13}$C, $\delta^{15}$N and $\delta^{34}$S values ranged from $-20.6‰$ to $-18.9‰$ (mean $= -19.5 \pm 1.0‰$), $+6.5‰$ to $+9.7‰$ (mean $= +8.5 \pm 1.7‰$), and $+3.7‰$ to $+8.5‰$ (mean $= +5.3 \pm 2.7‰$), respectively. GUsi-1110, GUsi-1111 and GUsi-1113 yielded radiocarbon ages of $1593 \pm 28$ $^{14}$C yr. BP, $1552 \pm 29$ $^{14}$C yr. BP, and $1431 \pm 29$ $^{14}$C yr. BP, respectively, giving an average date of death between AD 412–656.

Arctic fox bone ($n = 3$) $\delta^{13}$C, $\delta^{15}$N and $\delta^{34}$S values ranged from $-15.8‰$ to $-13.4‰$ (mean $= -14.9 \pm 1.3‰$), $+7.8‰$ to $+10.7‰$ (mean $= +9.0 \pm 1.5‰$), and $+0.6‰$ to $+1.9‰$ (mean $= +1.4 \pm 0.7‰$), respectively. GUsi-2118 and GUsi-2126 yielded radiocarbon ages of $2605 \pm 30$ $^{14}$C yr. BP and $2160 \pm 30$ $^{14}$C yr. BP, giving an average date of death between 827–107 BC.

### 4.6. Birds

$\delta^{13}$C, $\delta^{15}$N and $\delta^{34}$S stable isotope analysis on eleven birds of varying breed were undertaken. Chicken ($n = 1$), duck ($n = 2$), tufted duck ($n = 1$), mallard ($n = 2$), common scoter ($n = 1$), swan ($n = 3$) and swan/goose ($n = 1$) bones gave $\delta^{13}$C, $\delta^{15}$N and $\delta^{34}$S values that ranged from $-21.3‰$ to $-6.9‰$ (mean $= -13.6 \pm 4.2‰$), $+1.9‰$ to $+16.1‰$ (mean $= +6.5 \pm 3.9‰$), and $-5.3‰$ to $+13.6‰$ (mean $= +3.0 \pm 5.0‰$), respectively.

### 4.7. Summary: isotopic and elemental measurements

Terrestrial herbivores had average $\delta^{13}C$, $\delta^{15}N$ and $\delta^{34}S$ values of $-21.3 \pm 0.4‰$, $+3.0 \pm 1.3‰$ and $+5.6 \pm 2.8‰$, respectively. Freshwater fish yielded average $\delta^{13}C$, $\delta^{15}N$ and $\delta^{34}S$ values of $-9.8 \pm 0.6‰$, $+5.9 \pm 0.6‰$ and $-2.7 \pm 1.4‰$, respectively, whilst marine mammals and fish had average $\delta^{13}C$, $\delta^{15}N$ and $\delta^{34}S$ values of $-14.7 \pm 0.7‰$, $+13.2 \pm 0.7‰$ and $+15.9 \pm 1.5‰$, respectively (Table 2). The results demonstrate that there is a clear distinction between the $\delta^{34}S$ values of terrestrial, freshwater and marine species (Fig. 4), and at $2\sigma$, the average $\delta^{13}C$, $\delta^{15}N$ and $\delta^{34}S$ values of the three food groups are all significantly different.

The average atomic C:S and N:S ratios, as well as average %S values for all mammalian, bird and fish samples analysed fall within the criteria set out by Nehlich and Richards (2009) to assess the quality of archaeological bone collagen for sulphur isotope analysis (Table 3). Combined mammalian, bird and fish C:S and N:S atomic ratios are also presented in Table 3. Mammalian and bird C:S atomic ratios averaged $528 \pm 123$ and N:S atomic ratios averaged $160 \pm 37$, whilst collectively, fish samples had an average C:S atomic ratio of $188 \pm 14$ and an average N:S atomic ratio of $56 \pm 5$. Although a broader range of animal species were analysed in Nehlich and Richards study which may account for the larger error range, the results here exhibit a smaller range and are more in keeping with the mammalian ranges presented by Richards et al. (2001) (C:S = $463 \pm 176$) and Craig et al. (2006) (C:S = $496 \pm 39$, N:S = $148 \pm 12$), and the fish ranges shown by Privat et al. (2007) (C:S = $198 \pm 28$, N:S = $61 \pm 8$).

## 5. DISCUSSION

### 5.1. Domestic animals: evidence of husbandry practices via stable isotope analysis

$\delta^{13}C$ and $\delta^{15}N$ values confirm that cows, caprines and horses were consuming a wholly terrestrial $C_3$ plant diet, however, $\delta^{34}S$ values varied by $14.9‰$ ($-1.0‰$ to $+13.9‰$, mean = $+5.6 \pm 2.8‰$), implying that these animals were acquiring their food from different geographical areas. There are currently no published $\delta^{34}S$ values for the vegetation surrounding Lake Mývatn, however, local flora can source its sulphur from three main reservoirs: rock sulphide (Mývatn $\delta^{34}S$ value: $-2.0‰$ to $+4.2‰$, (Torssander, 1989)), atmospheric $SO_2$ (Mývatn $\delta^{34}S$ value: $-1.8‰$ to $+3.4‰$, (Torssander, 1988)) and river, ground, and spring water in the area. Whilst $\delta^{34}S$ values for Mývatn water sup-

plies have not yet been established, previous studies have shown that magmatic sulphate from volcanic eruptions have the potential to influence local water sources, and sulphate $\delta^{34}S$ values have been found to range between $-1.7‰$ and $+10.1‰$ (Gíslason et al., 2002; Gíslason and Torssander, 2006; Robinson et al., 2009; Holm et al., 2010). Therefore, it is conceivable that the $\delta^{34}S$ value of sulphur in the Mývatn region could range from $-2.0‰$ to $+10.1‰$, and since plants are depleted in $^{34}S$ by $\sim1.5‰$ relative to their sulphate source (Trust and Fry, 1992), vegetation in the region is likely to have a $\delta^{34}S$ value that can vary between $-3.5‰$ and $+8.6‰$. Similarly, isotopic fractionation of sulphur in mammals is small relative to their diet (Peterson et al., 1985; Richards et al., 2003), and hence domestic animals raised in Mývatn and consuming local vegetation would not be expected to display a $\delta^{34}S$ value greater than $\sim+10‰$, however, this cut off value remains ambiguous until $\delta^{34}S$ values of flora and water samples in the area have been measured.

Two late medieval to early modern cows provided very similar $\delta^{13}C$ and $\delta^{15}N$ values (GU-20231: $-21.6‰$ and $+4.0‰$, respectively and GU-20241: $-21.9‰$ and $+4.1‰$, respectively), and it could mistakenly be assumed that these animals were reared in close proximity to each other, yet their $\delta^{34}S$ values tell a very different story ($+0.6‰$ vs. $+9.0‰$) (Fig. 5). Norse communities were known to participate in co-operative farming and animals were often moved around multiple farmsteads or jointly supervised uplands (e.g. Dugmore et al., 2012). The lower $\delta^{34}S$ value for GU-20231 indicates that this cow was likely grazing on vegetation that assimilated its sulphur predominantly from $\delta^{34}S$-depleted rock sulphide, whilst it is possible that animals with higher $\delta^{34}S$ values, as observed with GU-20241, were perhaps grazing closer to $\delta^{34}S$-enriched geothermal water sources or were reared on sea-spray effected coastal vegetation (see Section 5.2. for further discussion). A weak linear relationship ($R^2 = 0.24$) between $\delta^{15}N$ and $\delta^{34}S$ exists for cows, with animals that have a more enriched $\delta^{15}N$ value tending to have a depleted $\delta^{34}S$ value (Fig. 6A). Cattle grazing near Lake Mývatn are likely to have consumed plant material that was enriched in $^{15}N$ due to the decomposition of chironomid midges, which transports nitrogen from the lake to the shore, whilst for cows foraging further afield, their $\delta^{15}N$ values may be less enriched due to a decreasing effect of chironomid numbers with increasing distance from the shore (Gratton et al., 2008). Alternatively, animals being farmed closer to Lake Mývatn may have elevated $\delta^{15}N$ values due to the soil being fertilised with manure (Bogaard et al., 2007; Fraser et al., 2011).

Although the average $\delta^{13}C$ and $\delta^{15}N$ values for cattle and caprines are very similar ($-21.5‰$ vs. $-21.2‰$ and $+3.9‰$ vs. $+2.5‰$), their average $\delta^{34}S$ values are slightly different ($+4.1‰$ vs. $+6.7‰$) (Fig. 4), suggesting differing diet and/or grazing areas between the two groups. If sheep and goats were grazing in the Krafla lava fields, then they may have been consuming moss and lichens that are capable of accumulating their sulphur directly from atmospheric $SO_2$. Given that atmospheric $SO_2$ is likely to have varied over time due to numerous volcanic eruptions, it is not inconceivable that the higher $\delta^{34}S$ value of caprines

Table 2
Mean and standard deviations ($1\sigma$) of terrestrial, freshwater and marine animal bone collagen from Skútustaðir, Iceland.

| Species group | N | $\delta^{34}S$ [‰] | $\delta^{13}C$ [‰] | $\delta^{15}N$ [‰] |
|---|---|---|---|---|
| Terrestrial | 85 | $5.6 \pm 2.8$ | $-21.3 \pm 0.4$ | $3.0 \pm 1.3$ |
| Freshwater | 12 | $-2.7 \pm 1.4$ | $-9.8 \pm 0.6$ | $5.9 \pm 0.6$ |
| Marine | 15 | $15.9 \pm 1.5$ | $-14.7 \pm 0.7$ | $13.2 \pm 0.7$ |

Table 3
Mean and standard deviations (1$\sigma$) of C:S and N:S ratios and %S of archaeological samples from Skútustaðir.

| Species | N | C:S ratio (ave.) | N:S ratio (ave.) | %S (ave.) | C:S ratio | N:S ratio | %S |
|---|---|---|---|---|---|---|---|
| Cow | 32 | 544 ± 127 | 164 ± 38 | 0.21 ± 0.05 | 503[*] | 158[*] | 0.25[*] |
| Sheep/goat | 48 | 545 ± 116 | 165 ± 35 | 0.21 ± 0.05 | 521[*] | 163[*] | 0.26[*] |
| Horse | 5 | 575 ± 125 | 172 ± 41 | 0.20 ± 0.03 | 540[*] | 166[*] | 0.23[*] |
| Pig | 3 | 589 ± 38 | 179 ± 13 | 0.17 ± 0.01 | 401[*] | 124[*] | 0.32[*] |
| Char/trout | 12 | 190 ± 11 | 55 ± 3 | 0.56 ± 0.04 | 180[*] | 55[*] | 0.64[*] |
| Cod/haddock | 9 | 186 ± 18 | 58 ± 7 | 0.48 ± 0.03 | 196[*] | 61[*] | 0.62[*] |
| Seal | 6 | 579 ± 33 | 175 ± 11 | 0.21 ± 0.05 | 472[*] | 148[*] | 0.26[*] |
| Birds | 11 | 397 ± 56 | 120 ± 16 | 0.29 ± 0.05 | 417[*] | 128[*] | 0.29[*] |
| Arctic fox | 3 | 408 ± 79 | 122 ± 25 | 0.25 ± 0.03 | N/A | N/A | N/A |
| All mammals and birds | 108 | 528 ± 123 | 160 ± 37 | 0.22 ± 0.05 | 600 ± 300[*] | 200 ± 100[*] | 0.15–0.35[*] |
| All fish | 21 | 188 ± 14 | 56 ± 5 | 0.53 ± 0.05 | 175 ± 50[*] | 60 ± 20[*] | 0.4–0.8[*] |

[*] Nehlich and Richards (2009).
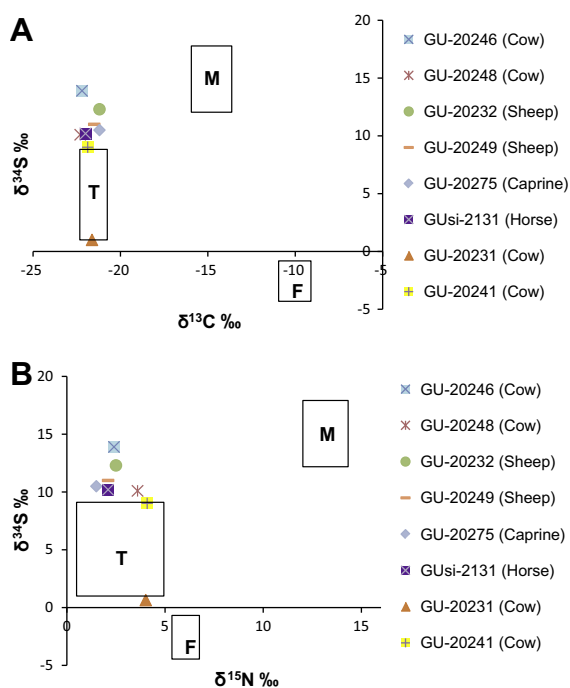


Fig. 5. Plots of $\delta^{13}C$ vs. $\delta^{34}S$ (A) and $\delta^{15}N$ vs. $\delta^{34}S$ (B) of Skútustaðir animals that lie outside the boundaries of terrestrial (T), marine (M) and freshwater (F) species. The figure also highlights the two cows, GU-20231 and GU-20241, with contrasting $\delta^{34}S$ values.
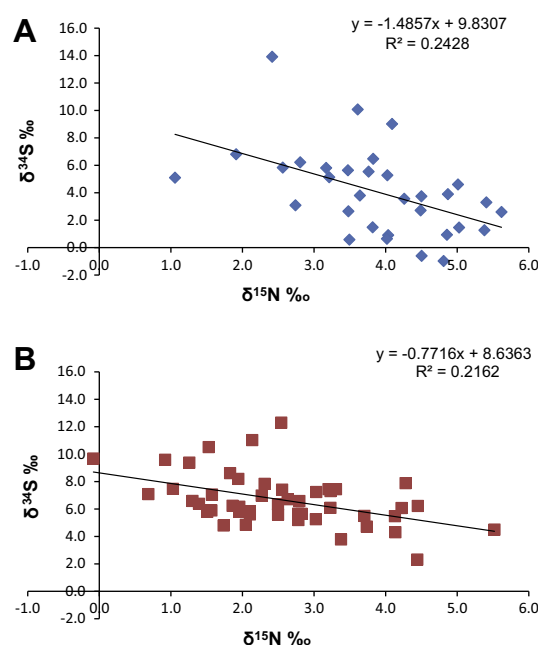


Fig. 6. Cow bone collagen (A) and caprine bone collagen (B) stable isotope values for archaeological samples from Skútustaðir. In both graphs a significant ($p < 0.01$) linear relationship is observed between increasing $\delta^{15}N$ and decreasing $\delta^{34}S$ values.

compared to cattle may be due to the consumption of a different food source with a more enriched isotopic signature. As observed with cows, there is a weak correlation ($R^2 = 0.22$) between $\delta^{15}N$ and $\delta^{34}S$, with caprines that have a more enriched $\delta^{15}N$ value tending to have depleted $\delta^{34}S$ value (Fig. 6B), suggesting that sheep and goats that were being kept closer to the lake were also consuming plants that had been $^{15}N$ enriched by chironomid midges.

Only five horses were sampled in this study and as expected their $\delta^{13}C$, $\delta^{15}N$ and $\delta^{34}S$ values were very similar to those observed in cattle and caprines. It is probable that, similarly to cattle, they were being managed closer to the farmstead at Skútustaðir rather than being grazed further afield. However, their average $\delta^{15}N$ value of +1.9‰ is less

enriched than cows, emphasising that the two species were likely being given different foodstuffs.

### 5.2. Domestic animals: evidence of regional trading

Haddock, cod and seal bones were all excavated from middens at Skútustaðir and demonstrate that although Lake Mývatn is approximately 50 km inland, established trade links to the coast were in place. The intake of marine resources was an important part of the Norse diet and in times when crop production or animal stocks were low, marine resources may have provided a major source of food (McGovern, *pers. comm.*). Whilst the boundary plots in Fig. 5 show that clear differences exist between terrestrial,

freshwater and marine species, two cows (GU-20246: $\delta^{34}$S +13.9‰ and GU-20248: $\delta^{34}$S +10.1‰), three sheep (GU-20232: $\delta^{34}$S +12.3‰, GU-20249: $\delta^{34}$S +11.0‰, and GU-20275: $\delta^{34}$S +10.5‰) and a horse (GUsi-2131: $\delta^{34}$S +10.2‰) displayed enriched $\delta^{34}$S values. The higher $\delta^{34}$S values may be attributed to the consumption of vegetation containing marine-derived sulphur, suggesting these animals may have been reared closer to the coast and trading of domestic animals was also occurring within Icelandic Norse communities. Studies have shown that sea-spray not only influences coastal soil $\delta^{34}$S values, as sulphate particles can be propelled inland over extensive distances (Zazzo et al., 2011), however, it is unlikely that the land around Lake Mývatn would have been affected by sea-spray as the $\delta^{34}$S values of these six animals are very distinct from the average $\delta^{34}$S values of their contemporaries.

The rate of bone collagen turnover is poorly understood, with estimates ranging from less than a year in birds (Hobson and Clark, 1992) to over ten years in adult humans (Hedges et al., 2007). Assuming that fully matured animals are slaughtered within a few years of being brought inland, their $^{34}$S signature is unlikely to have changed significantly from when they first arrived at Lake Mývatn. However, collagen turnover rates in juveniles should be higher than in adults, and therefore if young livestock grazing on coastal vegetation were then traded inland and reared for the remainder of their lives in the Lake Mývatn region, it is likely that their $\delta^{34}$S values would lie somewhere between a marine signature and the $\delta^{34}$S value for the local vegetation, and this may account for the intermediary $\delta^{34}$S values observed in some domestic animals.

### 5.3. Lake Mývatn birdlife: evidence of avian diet variability

The very broad range in $\delta^{13}$C (−21.3‰ to −6.9‰, mean = −13.6 ± 4.2‰), $\delta^{15}$N (+1.9‰ to +16.1‰, mean = +6.5 ± 3.9‰) and $\delta^{34}$S (−5.3‰ to +13.6‰, mean = −3.0 ± 5.0‰) values for birds reflects the variation in diet of each species. Analysis of modern detritus, algae, pondweed, larvae, zooplankton and mollusc samples taken from Lake Mývatn yielded $\delta^{13}$C values that varied between −22.6‰ and −10.1‰ and $\delta^{15}$N values that varied between −16.0‰ and +6.3‰ (Ascough et al., 2011). Freshwater resources would have been the main food supply for birds surrounding Lake Mývatn, and as eight of the eleven birds have a $\delta^{34}$S value below +4‰, this suggests that the waters of Lake Mývatn may have been their permanent home. Settlers brought the domestic fowl to Iceland in the 9th century, and whilst only one chicken has currently been analysed, its enriched $\delta^{13}$C, $\delta^{15}$N and depleted $\delta^{34}$S value (GUsi-2116: −18.3‰, +9.5‰ and +2.6‰, respectively) would suggest that it had consumed freshwater fish scraps. GUsi-2129's enriched $\delta^{15}$N value (+8.5‰) indicates that this duck may have also been consuming some animal protein. However, its $\delta^{34}$S value (+7.7‰) is midway between the values observed for a pure freshwater feeder and a pure marine feeder, which would suggest migratory birds also resided at Lake Mývatn. The enriched $\delta^{15}$N and $\delta^{34}$S values of GUsi-2130 (+16.0‰ and +13.6‰, respectively) demonstrate that although this bird spent the majority of its life

within a marine environment, it may have occasionally migrated to the fertile waters of Lake Mývatn. Alternatively, this bird could have spent its entire life at the coast, indicating that perhaps it was not just domestic animals, seals and marine fish that were being traded between communities.

### 5.4. $^{14}$C-dating: evidence of freshwater reservoir effects

$\delta^{13}$C and $\delta^{15}$N values for two of the three pigs examined in this study indicated they were consuming a variety of produce, including both terrestrial- and non-terrestrial-based resources (GUsi-1110: −18.9‰ and +9.7‰, respectively and GUsi-1111: −19.0‰ and +9.3‰, respectively). However, by solely considering their $\delta^{13}$C and $\delta^{15}$N values, there is no clear indication as to whether it was terrestrial animal, freshwater fish or marine fish/mammal protein that was being consumed. Given that the $\delta^{34}$S values of GUsi-1110 (+3.8‰) and GUsi-1111 (+3.7‰) are lower than the average $\delta^{34}$S values observed for the terrestrial animals (+5.6‰), and marine species recovered from the midden at Skútustaðir have an average $\delta^{34}$S value of +15.9‰, these results would suggest that the pigs have been consuming freshwater fish scraps, and the depleted $\delta^{34}$S values exhibited in trout and char bones (mean: −2.7 ± 1.4‰) corroborates this theory. Radiocarbon dating of GUsi-1110 (1593 ± 28 $^{14}$C yr. BP, cal. AD 412–540 (95.4% probability)) and GUsi-1111 (1552 ± 29 $^{14}$C yr. BP, cal. AD 426–573 (95.4% probability)) confirms that a large freshwater reservoir effect (FRE) was occurring as both pigs are significantly older than the landnám Viking settlement date of AD 871 ± 2 (Vésteinsson, 1998; McGovern et al., 2007; Einarsson and Aldred, 2011). It was assumed that GUsi-1113, with its slightly enriched $\delta^{13}$C (−20.6‰), $\delta^{15}$N (+6.5‰) and $\delta^{34}$S (8.5‰) values, may have been consuming a small proportion of terrestrial animal or marine resources, yet its radiocarbon date (1431 ± 29 $^{14}$C yr. BP) would suggest otherwise. As observed with GUsi-1110 and GUsi-1111, GUsi-1113 pre-dates the landnám and displayed an overall 2$\sigma$ calibrated age range of AD 576–656. This could suggest that this pig had been consuming freshwater resources from a different body of water to Lake Mývatn, and again potentially highlights that trading was taking place between Viking communities. Subsequently, human consumption of these pigs (and any other freshwater species) would influence their $^{14}$C ages and they too would appear older than their assigned cultural period (Ascough et al., 2007, 2010, 2011, 2012).

Arctic foxes are known to be opportunistic feeders that can adapt their diet depending on seasonal or geographical changes (Hersteinsson and MacDonald, 1996). They are renowned for preying on birds and stealing their eggs, and the large population of waterfowl surrounding Lake Mývatn would have provided an ample food supply for these predators. Even during the winter months, a considerable part of the lake and the Laxá River remains ice-free, allowing the foxes to hunt all year round. $\delta^{13}$C (mean: −14.9 ± 1.3‰), $\delta^{15}$N (mean: +9.0 ± 1.5‰) and in particular $\delta^{34}$S (mean: +1.4‰ ± 0.7‰) stable isotope analysis supports the theory that like pigs, they too may have been scavenging freshwater fish carcasses from the shores of the lake and river.

GUsi-2118 and GUsi-2126 were discovered just above the *landnám* tephra fall of AD 871 ± 2, yet $^{14}$C-dating estimates that these animals ranged from 2605 ± 30 $^{14}$C yr. BP (GUsi-2118) to 2160 ± 30 $^{14}$C yr. BP (GUsi-2126). These results offer an overall 2σ age range of 827–107 BC, which again is significantly earlier than the Viking settlement period and demonstrates that the $^{14}$C ages of these samples are affected by a freshwater reservoir effect (Ascough et al., 2007, 2010, 2011, 2012).

## 6. CONCLUSIONS

In the first comprehensive stable isotope study to be undertaken of archaeological fauna from one specific site within the Lake Mývatn area, utilisation of $^{34}$S analysis in conjunction with $^{13}$C and $^{15}$N analyses has revealed important details concerning the husbandry techniques and livestock trading practice of early Viking settlers in Iceland. This study is also the first instance in which sulphur isotope analysis has been carried out on animal remains from archaeological deposits in Iceland, and has proven to be a valuable tool for discriminating between terrestrial, freshwater and marine based diets.

Cattle bones were found to have lower $δ^{34}$S values but higher $δ^{15}$N values than the caprine bones analysed, which suggests that they were being kept closer to Lake Mývatn and perhaps feeding on $δ^{15}$N enriched grasses, whereas a more enriched $δ^{34}$S value for sheep and goat bones indicated they were grazing away from the lake and possibly consuming moss and lichens on the Krafla lava fields. Three 10th century pigs were analysed during this study and $δ^{13}$C and $δ^{15}$N values indicated that two of them were consuming a mixture of terrestrial and non-terrestrial resources, whilst $δ^{34}$S analyses and radiocarbon dating points towards the ingestion of freshwater fish as the non-terrestrial source. This suggests that early settlers allowed pigs to roam quite freely around the farmstead and consume domestic waste, or alternatively, if they were styed, they were deliberately being fed scraps that included non-terrestrial material.

It would be incorrect to assume that only the weathering of local bedrock dictates $δ^{34}$S results, as the majority of the terrestrial animals examined had a $δ^{34}$S value greater than +4.2‰, whilst almost all of the freshwater fish had $δ^{34}$S values less than −2.0‰. Iceland has been subjected to many volcanic eruptions before and since the *landnám*, and evidence has shown that the increased amounts of sulphurous magmatic gases have affected the $δ^{34}$S values of rivers, lakes and groundwater. The increase in sulphate concentration has led to an increase in $δ^{34}$S variability, and as a consequence, this variability has been transferred throughout the food chain. It is highly conceivable that the enriched sulphur isotope values observed in some terrestrial animals were the result of sea-spray affecting coastally reared animals, which were then moved inland.

Overall, there is an offset of ~8‰ in sulphur isotope values between terrestrial and freshwater systems in the Lake Mývatn region of Iceland, and the tight range for freshwater fish demonstrates the homogeneity of this environment with respect to $δ^{34}$S. Results have shown that the freshwater fish populating Lake Mývatn have a significantly different $δ^{34}$S value from their marine contemporaries, with the average $δ^{34}$S value of the two groups offset by approx. 18.5‰. As the remains of both freshwater and marine resources have been found in middens at Skútustaðir, sulphur isotope analysis will be a useful tool for reconstructing the diets of the human inhabitants, and while correcting for the freshwater $^{14}$C reservoir effect at Lake Mývatn is problematic, these results have nevertheless enabled us to differentiate whether the anomalously old $^{14}$C ages are due to a marine or freshwater $^{14}$C reservoir effect.

As a final point, further research into the use of sulphur isotopes in archaeology is required, but this study has demonstrated that if a system is well-defined, is investigated in a methodical manner and is supported with archaeological and palaeoenvironmental information, it is possible to use sulphur isotopes as an important tracer of diet.

## APPENDIX A. SUPPLEMENTARY DATA

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.gca.2013.07.008.

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