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Application of <sup>34</sup> 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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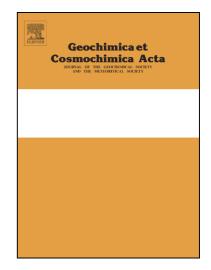
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- 1 Application of <sup>34</sup>S analysis for elucidating terrestrial, marine and freshwater
- 2 ecosystems: Evidence of animal movement/husbandry practices in an Early Viking
- 3 community around Lake Mývatn, Iceland
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### Abstract

3	Carbon and nitrogen stable isotope ratios ( $\delta^{12}$ C and $\delta^{12}$ N) have been used widely in
4	archaeology to investigate palaeodiet. Sulphur stable isotope ratios $(\delta^{34}S)$ have shown
5	great promise in this regard but the potential of this technique within archaeological
6	science has yet to be fully explored. Here we report $\delta^{34}S$ , $\delta^{13}C$ and $\delta^{15}N$ values for 129
7	samples of animal bone collagen from Skútustaðir, an early Viking age (landnám)
8	settlement in north-east Iceland. This dataset represents the most comprehensive study to
9	date of its kind on archaeological material and the results show a clear offset in $\delta^{34} S$
10	values between animals deriving their dietary resources from terrestrial (mean = $\pm$ 5.6 $\pm$
11	2.8%), freshwater (mean = -2.7 $\pm$ 1.4%) or marine (mean = +15.9 $\pm$ 1.5%) reservoirs
12	(with the three food groups being significantly different at $2\sigma$ ). This offset allows
13	reconstruction of the dietary history of domesticated herbivores and demonstrates
14	differences in husbandry practices and animal movement/trade, which would be
15	otherwise impossible using only $\delta^{13}C$ and $\delta^{15}N$ values. For example, several terrestrial
16	herbivores displayed enriched bone collagen $\delta^{34}$ S values compared to the geology of the
17	Lake Mývatn region, indicating they may have been affected by sea-spray whilst being
18	pastured closer to the coast, before being traded inland. Additionally, the combination of
19	heavy $\delta^{15}N$ values coupled with light $\delta^{34}S$ values within pig bone collagen suggests that
20	these omnivores were consuming freshwater fish as a significant portion of their diet.
21	Arctic foxes were also found to be consuming large quantities of freshwater resources and
22	radiocarbon dating of both the pigs and foxes confirmed previous studies showing that a
23	large freshwater radiocarbon (14C) reservoir effect exists within the lake. Overall, these
24	stable isotope and <sup>14</sup> C data have important implications for obtaining a fuller
25	reconstruction of the diets of the early Viking settlers in Iceland, and may allow a clearer
26	identification of the marine and/or freshwater <sup>14</sup> C reservoir effects that are known to exist
27	in human bone collagen.

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### Keywords

Stable isotopes, <sup>34</sup>S, <sup>13</sup>C, <sup>15</sup>N, palaeodiet, Iceland

### 1. Introduction

2	Extensive controlled feeding studies of modern terrestrial, freshwater and marine
3	species have shown that that diet can directly influence carbon $(\delta^{13}C)$ and nitrogen $(\delta^{15}N)$
4	stable isotope values in the tissues of the consumer (DeNiro and Epstein, 1978, 1981).
5	Archaeologists were very quick to exploit this theory and stable isotope analysis of
6	preserved bone collagen is now routinely utilised in palaeodietary reconstruction studies,
7	providing archaeologists with a detailed insight into prehistoric diet (Schoeninger et al.,
8	1983; Tauber, 1981; Van der Merwe and Vogel, 1978).
9	Recent advances in continuous-flow isotope-ratio mass spectrometry (CF-IRMS)
10	have allowed sulphur isotopes $(\delta^{34}S)$ to be measured from organic and inorganic materials
11	(Fritzsche and Tichomirowa, 2006; Fry, 2007; Fry et al., 1996; Giesemann et al., 1994;
12	Grassineau et al., 2001, 2006; Hansen et al., 2009; Yun et al., 2005) and over the past
13	decade there has been a marked increase in the use of sulphur isotopes in conjunction
14	with carbon and nitrogen isotopes to aid our understanding of the diet and movement of
15	prehistoric animals and humans (Craig et al., 2010; Howcroft et al., 2012; Hu et al., 2009;
16	Macko et al., 1999; Nehlich et al., 2012; Oelze et al., 2012a, 2012b; Vika, 2009; Richards
17	et al., 2001, 2003). Sulphur isotopes have also been exploited to explore the variability in
18	terrestrial-, marine- and freshwater-based diets (Craig et al., 2006; Lamb et al., 2012;
19	Nehlich et al., 2010, 2011; Privat et al., 2007), have been used as indicators of
20	environmental changes (Newton and Bottrell, 2007; Wadleigh, 2003; Yun et al., 2010)
21	and are regularly utilised in food authentication studies (Bahar et al., 2008; Osorio et al.,
22	2011; Rummel et al., 2010; Tanz and Schmidt, 2010).
23	Previous findings from the Lake Mývatn region of north-east Iceland have
24	demonstrated a significant overlap between the $\delta^{15} N$ values of both modern and
25	archaeological-age terrestrial herbivore bone collagen and the $\delta^{15} N$ values of modern and
26	archaeological-age freshwater fish. $\delta^{13} C$ values of freshwater biota were also found to be
27	similar to those of marine resources (Ascough et al, 2010), and consequently, separation
28	of herbivores, freshwater fish and marine fish as components of human diet is difficult
29	using only $\delta^{13} C$ and $\delta^{15} N$ analyses. Likewise, complications were encountered when
30	human bone collagen samples from the same region were radiocarbon (14C) dated, as the
31	exact proportion of freshwater and marine carbon consumed was indeterminable, which
32	prevented ages being corrected for both marine and freshwater <sup>14</sup> C reservoir effects

1	(Ascough et al. 2012). Freshwater and marine radiocarbon reservoir effects (FRE and
2	MRE, respectively) are <sup>14</sup> C age offsets between CO <sub>2</sub> in the atmosphere and the freshwater
3	or marine carbon reservoirs. The MRE arises because of the extended residence time of
4	carbon in the global marine reservoir, during which time radioactive decay of the <sup>14</sup> C
5	occurs. It has a global average value of approximately 400 <sup>14</sup> C years for surface waters
6	and can be corrected for in order to produce a truer calendar age range (Reimer et al.
7	2009). FREs are brought about by the input of low <sup>14</sup> C-activity carbon (e.g. carbon from
8	dissolution of geological carbonates or from high temperature geothermal water-rock
9	interactions), or restriction of atmosphere-water CO <sub>2</sub> exchange (e.g. via density
10	stratification or ice cover). Unlike the MRE, a universal amendment cannot be
11	implemented as the magnitude of FREs is site dependent and can fluctuate significantly
12	(Ascough et al., 2011; Keaveney and Reimer, 2012).
13	The purpose of this study was to utilise $\delta^{34}$ S stable isotope measurements in animal
14	and bird bone collagen from remains found in midden deposits at a Viking farmstead on
15	Lake Mývatn in an attempt to distinguish between terrestrial, marine and freshwater
16	dietary components. In turn, this could allow the identification of terrestrial, freshwater
17	and marine components in the human diet. To date, this is the first study of its kind to
18	examine sulphur isotopes in bone collagen from archaeological remains found in Iceland
19	It is also the largest, single-site sulphur isotope study to be undertaken, in which 129
20	bones of domesticated and wild fauna from Skútustaðir were analysed. These data were
21	produced, together with C and N stable isotope ratio measurements and <sup>14</sup> C age
22	measurements, as part of a multi-isotope approach, building on previous work to
23	reconstruct animal diet and human activity in the region (Ascough et al. 2007, 2010,
24	2012).
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#### 2. Background

2.1	Geographical	&	Historical	In	formation

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

#### 2.2 Geology of the Lake Mývatn Area

The area surrounding Lake Mývatn is volcanic in nature, with igneous rocks of the tholeitic series dominating the landscape. The series is split into three subsections: (1) basaltic rocks, which are most abundant, comprising of picrite, olivine tholeite and tholeite; (2) intermediate rocks which include icelandite and basaltic icelandite; and (3) silicic rocks which include dacite and rhyolite (Jakobsson 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

1 draining into the River Laxá in the west, the geothermal waters provide Lake Mývatn 2 with plentiful supplies of silica and sulphate, whilst cooler waters deliver phosphate to the lake (Kristmannsdóttir and Ármannsson, 2004). 3 4 2.3 Isotope Geochemistry of Iceland 5 6 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% 7 and +30% (Claypool et al, 1980; Strauss, 1997) and sulphate in marine water providing a 8 very isotopically uniform reservoir of  $\delta^{34}S = +21\%$  (Rees et al., 1978). In coastal 9 regions, sulphur-containing particles can be propelled inland in a process known as the 10 sea-spray effect, causing soil  $\delta^{34}$ S values to be similar to that of seawater (Wadleigh et al., 11 1994). 12 Intensive weathering of igneous rocks causes leached sulphides to filter into ground 13 14 and stream water systems, enabling plants to accumulate the oxidised sulphate form in their roots. Oxidation of pyrite (FeS<sub>2</sub>) produces negligible isotopic fractionation (Nakai 15 and Jensen, 1964; Taylor and Wheeler, 1984), whilst plant  $\delta^{34}$ S values are on average 16 1.5% depleted compared to their sulphate source (Trust and Fry, 1992). Analysis of total 17 sulphur in volcanic rocks from the Krafla-Námafjall fissure swarm neighbouring Lake 18 Mývatn provided  $\delta^{34}$ S values that ranged between -2.0% and +4.2% (mean: -0.8%) 19 (Torssander, 1989), whilst examination of transitional basaltic and rhyolitic rocks from 20 the Katla Volcanic Centre in southern Iceland gave similar  $\delta^{34}$ S values, ranging between -21 22 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 23 relatively small,  $\delta^{34}$ S values of Lake Mývatn flora should be similar to those reported by 24 25 Torssander (1989). However, Icelandic lava fields are sparsely vegetated with lichen and 26 moss (Bjarnason, 1991), which can accumulate their sulphur directly from atmospheric 27 sulphur dioxide (SO<sub>2</sub>) with very little isotopic fractionation (Krouse, 1977). SO<sub>2</sub> gas produced during the eruption of Krafla in July 1980 yielded  $\delta^{34}$ S values between -1.8% 28 and +3.4% (Torssander, 1988), however, it is conceivable that atmospheric SO<sub>2</sub> in 29 30 Iceland has varied with time due to the volcanic nature of the island. Wet deposition of 31 sulphate from the aqueous oxidation of atmospheric SO<sub>2</sub> 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

2 deposited has been shown to be negligible in areas where SO<sub>2</sub> emissions are high (Nriagu 3 and Coker, 1978), and compared to other parts of Iceland, the Mývatn region has little rain- or snowfall (Einarsson, 1979). 4 The eruption of the Grímsvötn volcano in 1996, which is situated below the 5 Vatnajökull Glacier in south-east Iceland, caused the Skeiðará River to flood, and water 6 7 samples taken before and during the early stages of the overflow revealed a post-eruption 8 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 9 10 gases were oxidised to sulphate upon dissolution in water (Gíslason et al., 2002). 11 Together with water samples taken from kettle-hole lakes that had formed since the 1996 12 glacier-outburst flood, further groundwater, geothermal spring water and Skeiðará River samples were retrieved between 1998 and 2001 from the Skeiðarársandur outwash plain 13 (Robinson et al., 2009).  $\delta^{34}$ S values from river sulphates ranged from +3.4% to +8.8%, 14 15 with the higher value again attributed to magmatic sulphate from the Grímsvötn caldera, 16 whilst the lower end value was measured during normal discharge; both values fall within 17 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 18 spring water had an average  $\delta^{34}$ S value of +4.1%. Water from the kettle-hole lakes was 19 found to be depleted in  $^{34}$ S, with  $\delta^{34}$ S values ranging between -1.7% and 0.0%, which 20 21 could be attributed to dissolved sulphate originating from igneous sulphide minerals. 22 Samples taken from the Hekla cold springs in southern Iceland, fifteen years after the 23 volcano last erupted, revealed that magmatic degassing into groundwater was still occurring. Dissolved sulphate in these water samples displayed  $\delta^{34}$ S values between 24 +1.5% and +4.3%, with magmatic sulphate estimated to have had a  $\delta^{34}$ S value of around 25 +7.0% (Holm et al., 2010). Thus, the numerous volcanic eruptions that have occurred 26 since Iceland was first settled are likely to have influenced the  $\delta^{34}$ S values of all water 27 sources feeding Lake Mývatn and its surrounding landscape. However, whilst Icelandic 28 rock and water samples have demonstrated  $\delta^{34}$ S values that span from -2% to +10, and 29 atmospheric SO<sub>2</sub>  $\delta^{34}$ S values in the Mývatn region may vary between -1.8% and +3.4%, 30 what is evident is that they are all isotopically very distinct from the  $\delta^{34}$ S value of 31 32 seawater (Figure 2).

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2	2.4 Isotope Biochemistry of Iceland
3	$\delta^{13}\text{C}$ analysis of various plants and lichens from four lakes in northern Iceland
4	provided values ranging between -30.9% and -23.3% (Wang and Wooller, 2006).
5	Likewise, the analysis of moss, grass, willow and liverwort samples from the geothermal
6	area of Kerlingarfjöll in central Iceland produced $\delta^{13}C$ values between -28.8% $\epsilon$ and -
7	$20.4\%$ (Skrzypek et al., 2008). Ascough et al. (in press) measured $\delta^{13}$ C on a variety of
8	modern flora from four sites close to Lake Mývatn and at one location approximately 5
9	km to the west of the lake, and found that the vegetation ranged from -31.6% to -26.9%.
10	These results are within the expected range for plants in the northern hemisphere
11	following a C <sub>3</sub> photosynthetic pathway, with the more enriched value of -20.4% from
12	Kerlingarfjöll being attributed to the moss growing in a colder climate (Skrzypek et al.,
13	2007). Aquatic plant samples from one site had $\delta^{13}C$ values averaging -13.3%, which is
14	characteristic of freshwater plants in Iceland (Wang and Wooller, 2006). Previous stable
15	isotope studies of archaeological bone samples discovered at various sites surrounding
16	Lake Mývatn indicate that $\delta^{13}C$ values for terrestrial animals ranged from -22.1% to -
17	$20.3\%$ o, whilst modern and archaeological freshwater fish displayed $\delta^{13}C$ values from -
18	$16.0\%$ to -7.9%. Omnivorous pigs and various birds displayed a large range of $\delta^{13}C$
19	values (-22.5% to -16.9% and -24.8% to -7.9%, respectively), reflecting the mixed
20	terrestrial, freshwater and possibly marine diet they would have been consuming
21	(Ascough et al., 2007, 2010, 2012, in press). Although cod and haddock bones have been
22	recovered at Skútustaðir, prior to this study, no stable isotope analysis had been
23	undertaken on these samples. However, cod from four archaeological sites in the north-
24	east Atlantic yielded $\delta^{13}C$ values between -14.7% and -11.3% (Barrett, 2008, 2011;
25	Russell, 2011).
26	It is well established that, although <sup>13</sup> C trophic level shifts are small, <sup>15</sup> N values have
27	been shown to shift between $+3\%$ and $+5\%$ with each trophic level in marine and
28	terrestrial food chains (Schoeninger and DeNiro, 1984). Given that terrestrial plants have
29	$\delta^{15}$ N values ranging between approximately 0 and +5%, herbivores and carnivores should
30	accordingly exhibit $\delta^{15}$ N values of ~+4 to +9% $_{e}$ and ~+8 to +13% $_{e}$ , respectively. Within
31	the marine environment, $\delta^{15}$ N values can range between ~+15 to +20% as a consequence
32	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,

1 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%) 2 to +9.7%) (Mateo et al., 2008). Freshwater fish have also displayed enriched  $\delta^{15}$ N 3 values; however studies have shown that some species have  $\delta^{13}$ C and  $\delta^{15}$ N values that are 4 similar to those observed within a terrestrial environment (Dufour et al., 1999). 5 6 Therefore, the introduction of a third stable isotope, namely sulphur, to aid in the 7 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 8 9 recovered from the four sites mentioned previously in northern Iceland and found that 10 values ranged from -12.4% to +5.6%, with many of the samples having values lower than -6.0%. Similarly, the analysis of moss, grass, willow and liverwort specimens from 11 Kerlingarfjöll produced  $\delta^{15}$ N values between -5.5% and -1.7% (Skrzypek et al., 2008). 12 Ascough et al. (in press) determined that modern flora from around Lake Mývatn had 13  $\delta^{15}$ N values between -9.1% and +6.5%. It has been noted that in soils depleted in 14 15 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. 16 (2007, 2010, 2012, in press) found that  $\delta^{15}N$  values for modern and archaeological 17 terrestrial animals ranged between -1.5% and +5.9%, whilst freshwater fish displayed 18  $\delta^{15}$ N values from +3.1% to +8.5%, and north-east Atlantic cod yielded  $\delta^{15}$ N values from 19 +11.9% to +15.4% (Barrett, 2008, 2011; Russell, 2011). Again, the  $\delta^{15}$ N values for pigs 20 21 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 22 ranges for Icelandic terrestrial and aquatic plants and fauna from the Lake Mývatn region 23 is illustrated in Figure 3. 24 Oceanic primary producers demonstrate sulphate  $\delta^{34}$ S values between +17% and 25 +21% (Peterson and Fry, 1987), however,  $\delta^{34}$ S values for organisms living within a 26 27 freshwater environment have been shown to vary anywhere between -22% and +20% 28 due to differing sulphur sources in the local geology as well as anaerobic bacteria residing 29 within lakes and rivers, which can reduce sulphate ions to hydrogen sulphide (H<sub>2</sub>S) (Faure, 1977; Peterson and Fry, 1987). For mammals to successfully thrive, sulphur-30 31 containing biochemical compounds, such as the essential amino acids, methionine and 32 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.,
- 2 1985; Richards et al., 2003). Therefore, consumers eating produce from within the Lake
- 3 Mývatn vicinity should have similar  $\delta^{34}$ S values to the surrounding terrestrial vegetation.
- 4 Currently, there are no published  $\delta^{34}$ S values for either animal or human archaeological
- 5 remains from Iceland, and globally only a small number of sulphur isotope studies have
- 6 been undertaken on archaeological material. However, this is now changing rapidly and
- 7 more recently an increasing number of archaeological studies have utilised sulphur
- 8 isotope analysis as part of their investigations (Nehlich et al., 2011; Privat et al., 2007;
- 9 Vika, 2009). 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 (Hu et al., 2009; Nehlich et al., 2010; Richards et
- 12 al., 2001).

1	
2	3. Methodology
3	3.1 Sampling location and materials
4	Skútustaðir, situated on the southern shore of Lake Mývatn (65° 57′ N 17° 03′ W),
5	was excavated during the NSF-funded IPY program "Long Term Human Ecodynamics in
6	the Norse North Atlantic: cases of sustainability, survival, and collapse". After the
7	discovery of an archaeological midden in 2007, the site was explored in greater detail
8	during a series of excavations between 2008 and 2010 (Edwald and McGovern, 2009,
9	2010; Hicks, 2010; Hicks et al., 2009; Hicks and Pálsdóttir, 2011), and a large number of
10	animal bones and artefacts were discovered. The midden site is on a natural rise in the
11	landscape, and due to the porous nature of the bedrock, drainage conditions are good and
12	no pockets of waterlogged sediments were noted. The pH of the soil is around 6.5,
13	providing favourable conditions for preservation of animal bones. The work discussed in
14	this paper deals solely with excavations carried out during the 2008 and 2009 field trips
15	and a list of all samples analysed with their stratigraphic context and chronology is
16	presented in the supplementary data section.
17	
18	3.2 Extraction of Bone Collagen
19	A modified version of the Longin method was used to extract the collagen
20	component from the animal bones (Longin, 1971). Sample surfaces were initially cleaned
21	using a Dremel® multi-tool, before they were lightly crushed into smaller fragments and
22	immersed in 1M HCl for approx. 24 h to effect demineralisation. The acid was then
23	decanted and samples were rinsed with ultra-pure water to remove any remaining
24	dissociated carbonates, acid soluble contaminants and solubilised bioapatite. The
25	gelatinous-like material was heated gently to ~80°C in ultra-pure water to denature and
26	solubilise the collagen. After cooling, the solution was filtered, reduced to approx. 5 ml

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and freeze-dried.

3.3 Carbon, Nitrogen and Sulphur Isotope Ratio Analyses

1	$\delta^{13}$ C, $\delta^{15}$ N and $\delta^{34}$ S stable isotope measurements were obtained using a continuous-
2	flow isotope ratio mass spectrometer (Thermo Scientific Delta V Advantage (Bremen,
3	Germany)) coupled to a Costech ECS 4010 elemental analyser (EA) (Milan, Italy) fitted
4	with a pneumatic autosampler. Samples were weighed into tin capsules ( $\sim$ 600 $\mu$ g for
5	$\delta^{13}C$ 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
6	ways. The EA was coupled to the mass spectrometer via a ConfloIII <sup>TM</sup> and samples were
7	combusted in a reactor containing chromium oxide and silvered cobaltous/cobaltic oxide
8	at $1020^{\circ}$ C to produce $N_2$ and $CO_2$ . The gases were then passed over a reduction reactor
9	containing reduced copper wires at 650°C. A magnesium perchlorate trap was used to
10	eliminate water produced during the combustion process and the gases were separated in
11	a 3 m stainless steel Porapak QS 50-80 mesh GC column heated to 45°C. Alternatively,
12	the EA was coupled via a ConfloIV <sup>TM</sup> and samples were combusted in a single reactor
13	containing tungstic oxide and copper wires at $1020^{\circ}\text{C}$ to produce $N_2$ and $CO_2$ . The gases
14	were then separated in a 2 m stainless steel Porapak QS 50-80 mesh GC column heated to
15	$70^{\circ}$ C. The latter system was used to obtain $\delta^{34}$ S values and the column was heated to
16	90°C to separate SO <sub>2</sub> . Helium (100mL/min) was used as a carrier gas throughout the
17	procedure. N2, CO2 and SO2 entered the mass spectrometer via an open split arrangement
18	within the ConfloIII <sup>TM</sup> /ConfloIV <sup>TM</sup> and were analysed against their corresponding
19	reference gases.
20	For every ten unknown samples, in-house gelatine standards, which are calibrated to
21	the international reference materials USGS40 (-26.39%o), USGS41 (+37.63%o), IAEA-
22	CH-6 (-10.45%), USGS25 (-30.41%), IAEA-N-1 (+0.43%) and IAEA-N-2 (+20.41%),
23	were run in duplicate. Results are reported as per mil (%) relative to the internationally
24	accepted standards VPDB and AIR with $1\sigma$ precisions of $\pm$ 0.2% and $\pm$ 0.3% for $\delta^{13}C$
25	and $\delta^{15}N$ , respectively. All animals analysed had C:N atomic ratios that fell within the
26	range of 2.9 to 3.6, indicating good bone collagen preservation (DeNiro, 1985). For $\delta^{34}S$
27	analysis, two internal standards, which are calibrated to the international reference
28	materials IAEA-S-1 (-0.3%o), IAEA-S-3 (-32.55%o) and IAEA-S-4 (+16.90%o), were run
29	for every five unknown samples. Results are reported as per mil (%o) relative to the
30	internationally accepted standard VCDT and the precision was $\pm 0.6\%$ o. 25% of $\delta^{34}$ S
31	analyses were carried out in duplicate; there were no significant differences in the
32	reproducibility of the results.
33	Nehlich and Richards (2009) analysed a variety of mammalian and fish bone
34	archaeological samples, with the objective of introducing quality control standards for

1	measuring sulphur isotopes in bone collagen. They found that, on average, mammalian	
2	and bird bone collagen had an atomic C:S ratio of $600 \pm 300$ , an atomic N:S ratio of 20	
3	± 100 and contained between 0.15 and 0.35% sulphur, whilst fish bone collagen was	
4	found to have an atomic C:S ratio of 175 $\pm$ 50, an atomic N:S ratio of 60 $\pm$ 20 and	
5	contained between 0.4 and 0.8% sulphur. A small number of individual samples in this	
6	study fell outside the above ranges and are excluded from the discussion (shown in italics	
7	and bold italics in their respective data tables in Supplementary Data Section). Two	
8	samples (highlighted by an asterisk in their respective data tables) have still been included	
9	as their values fall within the desired ranges when corrected for weighing errors on the	
10	analytical balance.	
11		
12	3.4 Radiocarbon Dating	
13	Radiocarbon ages were obtained from selected samples in this study. CO2 was	
14	generated from collagen via combustion following the method of Vandeputte et al.	
15	(1996). Following cryogenic purification, $\delta^{13}C$ was measured on an aliquot of the $CO_2$	
16	for normalization of sample <sup>14</sup> C/ <sup>13</sup> C ratios. This was achieved on a VG SIRA 10 isotope	
17	ratio mass spectrometer, using NBS 22 (oil) and NBS 19 (marble) as standards. The	
18	method of Slota et al. (1987) was used to convert a 3 ml aliquot of the CO <sub>2</sub> to graphite for	
19	<sup>14</sup> C measurement by accelerator mass spectrometry (AMS). Sample <sup>14</sup> C/ <sup>13</sup> C ratios were	
20	measured with carbon in the +1 charge state on the SUERC SSAMS at 245 keV.	
21	Calibrated age ranges at $2\sigma$ were obtained from sample $^{14}C$ ages using the atmospheric	
22	IntCal09 curve (Reimer et al., 2009) and OxCal version 4.1 (Bronk Ramsey 1995; 2001).	
23		
	IntCal09 curve (Reimer et al., 2009) and OxCal version 4.1 (Bronk Ramsey 1995; 2001).	
Y		

1 2 4. Results A summary of the stable isotope results is presented in Table 1 and plotted in Figure 3 4. All stable isotope and radiocarbon age measurements together with sampling areas and 4 stratigraphic contexts analysed in this study, can be found in Tables S1-S9 in the 5 6 Supplementary Data Section. 7 4.1 Terrestrial Herbivores 8 Cows (n=32):  $\delta^{13}$ C values ranged from -22.5 to -20.6% (mean = -21.5 ± 0.4%),  $\delta^{15}$ N 9 values from +1.1 to +5.6% (mean = +3.9  $\pm$  1.0%) and  $\delta^{34}$ S values from -1.0 to +13.9% 10 (mean =  $\pm 4.1 \pm 3.2\%$ ), respectively. 11 Caprines (sheep and goats) (n=48):  $\delta^{13}$ C values ranged from -22.0 to -20.4% (mean 12 = -21.2  $\pm$  0.4%),  $\delta^{15}$ N values from -0.1 to +5.5% (mean = +2.5  $\pm$  1.1%) and  $\delta^{34}$ S values 13 from +2.3 to +12.3% (mean  $= +6.7 \pm 1.9\%$ ). 14 Horses (n=5):  $\delta^{13}$ C values ranged from -22.4 to -21.4% (mean = -21.8 ± 0.4%),  $\delta^{15}$ N 15 values from +0.6 to +3.6% (mean = +1.9  $\pm$  1.3%), and  $\delta^{34}$ S values from +1.4 to +10.2% 16  $(\text{mean} = +5.7 \pm 3.2\%_0).$ 17 18 4.2 Freshwater Fish 19 The analysis of trout (n= 5) and char (n=7) bones yielded  $\delta^{13}$ C values that ranged 20 from -9.8 to -9.3% (mean = -9.6  $\pm$  0.2%) and -11.4 to -9.1% (mean = -10.0  $\pm$  0.8%), 21 respectively.  $\delta^{15}$ N values ranged from +5.0 to +6.8% (mean = +6.1 ± 0.7%) and +5.2 to 22 +6.8% (mean =  $+5.9 \pm 0.5\%$ ), respectively, whilst  $\delta^{34}$ S values ranged from -4.2 to -0.2% 23  $(\text{mean} = -2.4 \pm 1.5\%_0)$  and -4.3 to  $-0.4\%_0$   $(\text{mean} = -3.0 \pm 1.3\%_0)$ , respectively. 24 25

- 26 4.3 Marine Fish
- Haddock (n = 3) and cod bones (n=6) provided  $\delta^{13}$ C values from -14.6 to -14.0%
- 28 (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 ± 0.3%), and +13.3 to +14.5% 1 (mean +13.9  $\pm$  0.5%), respectively, whilst  $\delta^{34}$ S values varied from +12.4 to +15.9% 2  $(\text{mean} = +14.0 \pm 1.8\%)$ , and +15.6 and +17.5%  $(\text{mean} = +16.8 \pm 0.9\%)$ , respectively. 3 Haddock bones were on average 1.3% less enriched in nitrogen and 2.8% less enriched 4 in sulphur than cod bones. Although both fish are carnivores, adult cod are slightly 5 higher in the marine food web than haddock, and they have been known to eat smaller 6 cod, hence the difference in  $\delta^{15}N$  and  $\delta^{34}S$  values. 7 8 4.4 Marine Mammals 9 Seal bones (n=6)  $\delta^{13}$ C values ranged from -16.3 to -14.8% (mean = -15.3 ± 0.5%), 10  $\delta^{15}$ N values from +12.1 to +13.3% (mean = +12.7 ± 0.5%) and  $\delta^{34}$ S values from +14.3 to 11 +16.8% (mean =  $+15.9 \pm 1.0\%$ ). 12 13 4.5 Omnivorous Mammals 14 Pig bone (n=3)  $\delta^{13}$ C,  $\delta^{15}$ N and  $\delta^{34}$ S values ranged from -20.6 to -18.9% $_{0}$  (mean = -15  $19.5 \pm 1.0\%$ ), +6.5 to +9.7% (mean = +8.5 ± 1.7%), and +3.7 to +8.5% (mean = +5.3 ± 16 2.7‰), respectively. GUsi-1110, GUsi-1111 and GUsi-1113 yielded radiocarbon ages of 17  $1593 \pm 28^{-14}$ C yr. BP,  $1552 \pm 29^{-14}$ C yr. BP, and  $1431 \pm 29^{-14}$ C yr. BP, respectively, 18 19 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 20 =  $-14.9 \pm 1.3\%$ , +7.8 to +10.7% (mean =  $+9.0 \pm 1.5\%$ ), and +0.6 to +1.9% (mean = 21  $+1.4 \pm 0.7\%$ ), respectively. GUsi-2118 and GUsi-2126 yielded radiocarbon ages of 2605 22  $\pm 30^{14}$ C yr. BP and 2160  $\pm 30^{14}$ C yr. BP, giving an average date of death between 827-23 107 BC. 24 25 4.6 Birds 26  $\delta^{13}$ C,  $\delta^{15}$ N and  $\delta^{34}$ S stable isotope analysis on eleven birds of varying breed were 27 undertaken. Chicken (n=1), duck (n=2), tufted duck (n=1), mallard (n=2), common scoter 28 (n=1), swan (n=3) and swan/goose (n=1) bones gave  $\delta^{13}$ C,  $\delta^{15}$ N and  $\delta^{34}$ S values that 29

1 ranged from -21.3 to -6.9% (mean = -13.6  $\pm$  4.2%), +1.9 to +16.1% (mean = +6.5  $\pm$ 2 3.9%, and -5.3 to +13.6% (mean = +3.0 ± 5.0%), respectively. 3 4.7 Summary: Isotopic and Elemental Measurements 4 Terrestrial herbivores had average  $\delta^{13}$ C,  $\delta^{15}$ N and  $\delta^{34}$ S values of -21.3  $\pm$  0.4%, +3.0 5  $\pm 1.3\%$  and  $\pm 5.6 \pm 2.8\%$ , respectively. Freshwater fish yielded average  $\delta^{13}$ C,  $\delta^{15}$ N and 6  $\delta^{34}$ S values of -9.8 ± 0.6%, +5.9 ± 0.6% and -2.7 ± 1.4%, respectively, whilst marine 7 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‰ 8 and  $\pm 1.5\%$ , respectively (Table 2). The results demonstrate that there is a clear 9 distinction between the  $\delta^{34}$ S values of terrestrial, freshwater and marine species (Figure 10 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 11 12 significantly different. The average atomic C:S and N:S ratios, as well as average %S values for all 13 mammalian, bird and fish samples analysed fall within the criteria set out by Nehlich and 14 Richards (2009) to assess the quality of archaeological bone collagen for sulphur isotope 15 16 analysis (Table 3). Combined mammalian, bird and fish C:S and N:S atomic ratios are 17 also presented in Table 3. Mammalian and bird C:S atomic ratios averaged  $528 \pm 123$ 18 and N:S atomic ratios averaged  $160 \pm 37$ , whilst collectively, fish samples had an 19 average C:S atomic ratio of  $188 \pm 14$  and an average N:S atomic ratio of  $56 \pm 5$ . 20 Although a broader range of animal species were analysed in Nehlich and Richards study 21 which may account for the larger error range, the results here exhibit a smaller range and 22 are more in keeping with the mammalian ranges presented by Richards et al. (2001) (C:S 23  $= 463 \pm 176$ ) and Craig et al. (2006) (C:S =  $496 \pm 39$ , N:S =  $148 \pm 12$ ), and the fish 24 ranges shown by Privat et al. (2007) (C:S =  $198 \pm 28$ , N:S =  $61 \pm 8$ ).

1	
2	5. Discussion
3	5.1 Domestic Animals: Evidence of Husbandry Practices via stable isotope analysis
4	$\delta^{13} C$ and $\delta^{15} N$ values confirm that cows, caprines and horses were consuming a
5	wholly terrestrial $C_3$ plant diet, however, $\delta^{34}S$ values varied by 14.9% (-1.0 to +13.9%,
6	mean = $+5.6 \pm 2.8\%$ ), implying that these animals were acquiring their food from
7	different geographical areas. There are currently no published $\delta^{34}S$ values for the
8	vegetation surrounding Lake Mývatn, however, local flora can source its sulphur from
9	three main reservoirs: rock sulphide (Mývatn $\delta^{34}$ S value: -2.0 to +4.2‰, (Torssander,
10	1989)), atmospheric SO <sub>2</sub> (Mývatn $\delta^{34}$ S value: -1.8 to +3.4‰, (Torssander, 1988)) and
11	river, ground, and spring water in the area. Whilst $\delta^{34}S$ values for Mývatn water supplies
12	have not yet been established, previous studies have shown that magmatic sulphate from
13	volcanic eruptions have the potential to influence local water sources, and sulphate $\delta^{34}S$
14	values have been found to range between -1.7 and +10.1% (Gíslason and Torssander,
15	2006; Gíslason et al., 2002; Holm et al., 2010; Robinson et al., 2009). Therefore, it is
16	conceivable that the $\delta^{34}S$ value of sulphur in the Mývatn region could range from -2.0 to
17	+10.1%, and since plants are depleted in $^{34}$ S by ~1.5% relative to their sulphate source
18	(Trust and Fry, 1992), vegetation in the region is likely to have a $\delta^{34}$ S value that can vary
19	between -3.5% and +8.6%. Similarly, isotopic fractionation of sulphur in mammals is
20	small relative to their diet (Peterson et al, 1985, Richards et al., 2003), and hence
21	domestic animals raised in Mývatn and consuming local vegetation would not be
22	expected to display a $\delta^{34}$ S value greater than ~+10%, however, this cut off value remains
23	ambiguous until $\delta^{34}S$ values of flora and water samples in the area have been measured.
24	Two late medieval to early modern cows provided very similar $\delta^{13}C$ and $\delta^{15}N$ values
25	(GU-20231: -21.6% and +4.0%, respectively and GU-20241: -21.9% and +4.1%,
26	respectively), and it could mistakenly be assumed that these animals were reared in close
27	proximity to each other, yet their $\delta^{34}$ S values tell a very different story (+0.6% vs.
28	+9.0%) (Figure 5). Norse communities were known to participate in co-operative

25 (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‰) (Figure 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

1	observed with GU-20241, were perhaps grazing closer to $\delta^{34}$ S-enriched geothermal water
2	sources or were reared on sea-spray effected coastal vegetation (see Section 5.2 for
3	further discussion). A weak linear relationship ( $R^2 = 0.24$ ) between $\delta^{15}N$ and $\delta^{34}S$ exists
4	for cows, with animals that have a more enriched $\delta^{15}N$ value tending to have a depleted
5	$\delta^{34}$ S value (Figure 6A). Cattle grazing near Lake Mývatn are likely to have consumed
6	plant material that was enriched in <sup>15</sup> N due to the decomposition of chironomid midges,
7	which transports nitrogen from the lake to the shore, whilst for cows foraging further
8	afield, their $\delta^{15}N$ values may be less enriched due to a decreasing effect of chironomid
9	numbers with increasing distance from the shore (Gratton et al., 2008). Alternatively,
10	animals being farmed closer to Lake Mývatn may have elevated $\delta^{15}N$ values due to the
11	soil being fertilised with manure (Bogaard et al., 2007; Fraser et al., 2011).
12	Although the average $\delta^{13}C$ and $\delta^{15}N$ values for cattle and caprines are very similar (-
13	$21.5\% \textit{o}\ \textit{vs.}\ -21.2\% \textit{o}\ \text{and}\ +3.9\% \textit{o}\ \textit{vs.}\ +2.5\% \textit{o}),$ their average $\delta^{34}S$ values are slightly different
14	$(+4.1\% o\ vs.\ +6.7\% o)$ (Figure 4), suggesting differing diet and/or grazing areas between the
15	two groups. If sheep and goats were grazing in the Krafla lava fields, then they may have
16	been consuming moss and lichens that are capable of accumulating their sulphur directly
17	from atmospheric $SO_2$ . Given that atmospheric $SO_2$ is likely to have varied over time due
18	to numerous volcanic eruptions, it is not inconceivable that the higher $\delta^{34}S$ value of
19	caprines compared to cattle may be due to the consumption of a different food source
20	with a more enriched isotopic signature. As observed with cows, there is a weak
21	correlation ( $R^2 = 0.22$ ) between $\delta^{15}N$ and $\delta^{34}S$ , with caprines that have a more enriched
22	$\delta^{15}N$ value tending to have depleted $\delta^{34}S$ value (Figure 6B), suggesting that sheep and
23	goats that were being kept closer to the lake were also consuming plants that had been <sup>15</sup> N
24	enriched by chironomid midges.
25	Only five horses were sampled in this study and as expected their $\delta^{13}C$ , $\delta^{15}N$ and $\delta^{34}S$
26	values were very similar to those observed in cattle and caprines. It is probable that,
27	similarly to cattle, they were being managed closer to the farmstead at Skútustaðir rather
28	than being grazed further afield. However, their average $\delta^{15}N$ value of +1.9% $o$ is less
29	enriched than cows, emphasising that the two species were likely being given different
30	foodstuffs.
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5.2 Domestic Animals: Evidence of Regional Trading

1	Haddock, cod and seal bones were all excavated from middens at Skútustaðir and
2	demonstrate that although Lake Mývatn is approximately 50 km inland, established trade
3	links to the coast were in place. The intake of marine resources was an important part of
4	the Norse diet and in times when crop production or animal stocks were low, marine
5	resources may have provided a major source of food (McGovern, pers. comm.). Whilst
6	the boundary plots in Figure 5 show that clear differences exist between terrestrial,
7	freshwater and marine species, two cows (GU-20246: $\delta^{34}S$ +13.9% and GU-20248: $\delta^{34}S$
8	+10.1% $_{o}$ ), three sheep (GU-20232: $\delta^{34}$ S +12.3% $_{o}$ , GU-20249: $\delta^{34}$ S +11.0% $_{o}$ , and GU-
9	20275: $\delta^{34}S$ +10.5% $\epsilon$ ) and a horse (GUsi-2131: $\delta^{34}S$ +10.2% $\epsilon$ ) displayed enriched $\delta^{34}S$
10	values. The higher $\delta^{34}S$ values may be attributed to the consumption of vegetation
11	containing marine-derived sulphur, suggesting these animals may have been reared closer
12	to the coast and trading of domestic animals was also occurring within Icelandic Norse
13	communities. Studies have shown that sea-spray not only influences coastal soil $\delta^{34}S$
14	values, as sulphate particles can be propelled inland over extensive distances (Zazzo et
15	al., 2011), however, it is unlikely that the land around Lake Mývatn would have been
16	affected by sea-spray as the $\delta^{34}S$ values of these six animals are very distinct from the
17	average $\delta^{34}$ S values of their contemporaries.
18	The rate of bone collagen turnover is poorly understood, with estimates ranging from
19	less than a year in birds (Hobson and Clark, 1992) to over ten years in adult humans
20	(Hedges et al., 2007). Assuming that fully matured animals are slaughtered within a few
21	years of being brought inland, their <sup>34</sup> S signature is unlikely to have changed significantly
22	from when they first arrived at Lake Mývatn. However, collagen turnover rates in
23	juveniles should be higher than in adults, and therefore if young livestock grazing on
24	coastal vegetation were then traded inland and reared for the remainder of their lives in
25	the Lake Mývatn region, it is likely that their $\delta^{34} S$ values would lie somewhere between a
26	marine signature and the $\delta^{34}S$ value for the local vegetation, and this may account for the
27	intermediary $\delta^{34}S$ values observed in some domestic animals.
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20 /	
29	5.3 Lake Mývatn Birdlife: Evidence of Avian Diet Variability
30	The very broad range in $\delta^{13}$ C (-21.3 to -6.9%, mean = -13.6 ± 4.2%), $\delta^{15}$ N (+1.9 to
31	$+16.1\%$ , mean = $+6.5 \pm 3.9\%$ ) and $\delta^{34}S$ (-5.3 to +13.6%, mean = -3.0 $\pm$ 5.0%) values for
32	birds reflects the variation in diet of each species. Analysis of modern detritus, algae,

1	pondweed, larvae, zooplankton and mollusc samples taken from Lake Mývatn yielded
2	$\delta^{13}C$ values that varied between -22.6%e and -10.1%e and $\delta^{15}N$ values that varied between
3	-16.0% and +6.3% (Ascough et al., 2011). Freshwater resources would have been the
4	main food supply for birds surrounding Lake Mývatn, and as eight of the eleven birds
5	have a $\delta^{34}$ S value below +4‰, this suggests that the waters of Lake Mývatn may have
6	been their permanent home. Settlers brought the domestic fowl to Iceland in the 9 <sup>th</sup>
7	century, and whilst only one chicken has currently been analysed, its enriched $\delta^{13}C$ , $\delta^{15}N$
8	and depleted $\delta^{34}$ S value (GUsi-2116: -18.3%, +9.5% and +2.6%, respectively) would
9	suggest that it had consumed freshwater fish scraps. GUsi-2129's enriched $\delta^{15}N$ value
10	(+8.5%) indicates that this duck may have also been consuming some animal protein.
11	However, its $\delta^{34}$ S value (+7.7%) is midway between the values observed for a pure
12	freshwater feeder and a pure marine feeder, which would suggest migratory birds also
13	resided at Lake Mývatn. The enriched $\delta^{15}N$ and $\delta^{34}S$ values of GUsi-2130 (+16.0% and
14	+13.6%, respectively) demonstrate that although this bird spent the majority of its life
15	within a marine environment, it may have occasionally migrated to the fertile waters of
16	Lake Mývatn. Alternatively, this bird could have spent its entire life at the coast,
17	indicating that perhaps it was not just domestic animals, seals and marine fish that were
18	being traded between communities.
19	
19	
20	5.4 <sup>14</sup> C-dating: Evidence of Freshwater Reservoir Effects
21	$\delta^{13}C$ and $\delta^{15}N$ values for two of the three pigs examined in this study indicated they
22	were consuming a variety of produce, including both terrestrial- and non-terrestrial-based
23	resources (GUsi-1110: -18.9% and +9.7%, respectively and GUsi-1111: -19.0% and
24	+9.3%, respectively). However, by solely considering their $\delta^{13}C$ and $\delta^{15}N$ values, there is
25	no clear indication as to whether it was terrestrial animal, freshwater fish or marine
26	fish/mammal protein that was being consumed. Given that the $\delta^{34}S$ values of GUsi-1110
27	$(+3.8\%)$ and GUsi-1111 $(+3.7\%)$ are lower than the average $\delta^{34}$ S values observed for the
28	terrestrial animals $(+5.6\%e)$ , and marine species recovered from the midden at Skútustaðir
29	have an average $\delta^{34}$ S value of +15.9%, these results would suggest that the pigs have
30	been consuming freshwater fish scraps, and the depleted $\delta^{34}S$ values exhibited in trout
31	and char bones (mean: $-2.7 \pm 1.4\%$ ) corroborates this theory. Radiocarbon dating of
32	GUsi-1110 (1593 + $28^{-14}$ C vr BP cal AD 412-540 (95.4% probability)) and GUsi-1111

- 1  $(1552 \pm 29^{-14} \text{C yr. BP}, cal. \text{ AD } 426\text{-}573 (95.4\% \text{ probability})) \text{ confirms that a large}$
- 2 freshwater reservoir effect (FRE) was occurring as both pigs are significantly older than
- 3 the *landnám* Viking settlement date of AD 871 ± 2 (Vésteinsson, 1998; McGovern et al.,
- 4 2007; Einarsson and Aldred, 2011). It was assumed that GUsi-1113, with its slightly
- 5 enriched  $\delta^{13}$ C (-20.6%),  $\delta^{15}$ N (+6.5%) and  $\delta^{34}$ S (+8.5%) values, may have been
- 6 consuming a small proportion of terrestrial animal or marine resources, yet its
- radiocarbon date  $(1431 \pm 29^{14}\text{C yr. BP})$  would suggest otherwise. As observed with
- 8 GUsi-1110 and GUsi-1111, GUsi-1113 pre-dates the *landnám* and displayed an overall
- 9 2σ calibrated age range of AD 576-656. This could suggest that this pig had been
- 10 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 <sup>14</sup>C ages and they too would appear older than their assigned cultural
- 14 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
- 17 renowned for preying on birds and stealing their eggs, and the large population of
- 18 waterfowl surrounding Lake Mývatn would have provided an ample food supply for these
- 19 predators. Even during the winter months, a considerable part of the lake and the Laxá
- 20 River remains ice-free, allowing the foxes to hunt all year round.  $\delta^{13}$ C (mean: -14.9 ±
- 21 1.3%),  $\delta^{15}$ N (mean: +9.0 ± 1.5%) and in particular  $\delta^{34}$ S (mean: +1.4% ± 0.7%) stable
- 22 isotope analysis supports the theory that like pigs, they too may have been scavenging
- 23 freshwater fish carcasses from the shores of the lake and river. GUsi-2118 and GUsi-
- 24 2126 were discovered just above the *landnám* tephra fall of AD 871  $\pm$  2, yet <sup>14</sup>C-dating
- estimates that these animals ranged from  $2605 \pm 30^{-14}$ C yr. BP (GUsi-2118) to  $2160 \pm 30^{-14}$ C yr. BP (GUsi-2118) to  $2160 \pm 30^{-14}$ C yr.
- $^{14}$ C yr. BP (GUsi-2126). These results offer an overall  $2\sigma$  age range of 827-107 BC,
- which again is significantly earlier than the Viking settlement period and demonstrates
- that the <sup>14</sup>C ages of these samples are affected by a freshwater reservoir effect (Ascough
- 29 et al., 2007, 2010, 2011, 2012).

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#### 6. Conclusions

In the first comprehensive stable isotope study to be undertaken of archaeological 3 fauna from one specific site within the Lake Mývatn area, utilisation of <sup>34</sup>S analysis in 4 conjunction with <sup>13</sup>C and <sup>15</sup>N analyses has revealed important details concerning the 5 husbandry techniques and livestock trading practice of early Viking settlers in Iceland. 6 7 This study is also the first instance in which sulphur isotope analysis has been carried out 8 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. 9 Cattle bones were found to have lower  $\delta^{34}$ S values but higher  $\delta^{15}$ N values than the 10 caprine bones analysed, which suggests that they were being kept closer to Lake Mývatn 11 and perhaps feeding on  $\delta^{15}N$  enriched grasses, whereas a more enriched  $\delta^{34}S$  value for 12 13 sheep and goat bones indicated they were grazing away from the lake and possibly consuming moss and lichens on the Krafla lava fields. Three 10<sup>th</sup> century pigs were 14 analysed during this study and  $\delta^{13}$ C and  $\delta^{15}$ N values indicated that two of them were 15 consuming a mixture of terrestrial and non-terrestrial resources, whilst  $\delta^{34}S$  analyses and 16 17 radiocarbon dating points towards the ingestion of freshwater fish as the non-terrestrial 18 source. This suggests that early settlers allowed pigs to roam quite freely around the 19 farmstead and consume domestic waste, or alternatively, if they were styed, they were deliberately being fed scraps that included non-terrestrial material. 20 It would be incorrect to assume that only the weathering of local bedrock dictates 21  $\delta^{34}$ S results, as the majority of the terrestrial animals examined had a  $\delta^{34}$ S value greater 22 than +4.2%, whilst almost all of the freshwater fish had  $\delta^{34}$ S values less than -2.0%. 23 24 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 25 affected the  $\delta^{34}$ S values of rivers, lakes and groundwater. The increase in sulphate 26 concentration has led to an increase in  $\delta^{34}$ S variability, and as a consequence, this 27 28 variability has been transferred throughout the food chain. It is highly conceivable that 29 the enriched sulphur isotope values observed in some terrestrial animals were the result of 30 sea-spray affecting coastally reared animals, which were then moved inland.

Overall, there is an offset of  $\sim 8\%$  in sulphur isotope values between terrestrial and freshwater systems in the Lake Mývatn region of Iceland, and the tight range for

1	freshwater fish demonstrates the homogeneity of this environment with respect to $\delta^{34}$ S.
2	Results have shown that the freshwater fish populating Lake Mývatn have a significantly
3	different $\delta^{34}S$ value from their marine contemporaries, with the average $\delta^{34}S$ value of the
4	two groups offset by approx. 18.5%. As the remains of both freshwater and marine
5	resources have been found in middens at Skútustaðir, sulphur isotope analysis will be a
6	useful tool for reconstructing the diets of the human inhabitants, and while correcting for
7	the freshwater <sup>14</sup> C reservoir effect at Lake Mývatn is problematic, these results have
8	nevertheless enabled us to differentiate whether the anomalously old <sup>14</sup> C ages are due to a
9	marine or freshwater <sup>14</sup> C reservoir effect.
10	As a final point, further research into the use of sulphur isotopes in archaeology is
11	required, but this study has demonstrated that if a system is well-defined, is investigated
12	in a methodical manner and is supported with archaeological and palaeoenvironmental
13	information, it is possible to use sulphur isotopes as an important tracer of diet.
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#### References

- 3 Arnalds, Ó., Thorarinsdóttir, E.F., Metusalemsson, S., Johnsson, A., Gretarsson, E.,
- 4 Arnason, A. 1997. Soil Erosion in Iceland. Reykjavik: Icelandic SCS and the
- 5 Agricultural Research Institute.
- 6 Ascough, P.L., Cook, G.T., Church, M.J., Dugmore, A.J., McGovern, T.H., Dunbar, E.
- 7 Einarsson, Á., Friðriksson, A., Gestsdóttir, H. 2007. Reservoirs and radiocarbon: <sup>14</sup>C
- 8 dating problems in Mývatnssveit, northern Iceland. Radiocarbon 49, 947-961.
- 9 Ascough, P.L., Cook, G.T., Church, M.J., Dunbar, E., Einarsson Á., McGovern, T.H.,
- Dugmore, A.J., Perdikaris, S., Hastie, H., Friðriksson, A., Gestsdóttir, H. 2010.
- 11 Temporal and spatial variations in freshwater <sup>14</sup>C Reservoir Effects: Lake Mývatn,
- northern Iceland. Radiocarbon 52, 1098-1112.
- Ascough, P.L., Cook, G.T., Hastie, H., Dunbar, E., Church, M.J., Einarsson, Á.,
- 14 McGovern, T.H., Dugmore, A.J. 2011. An Icelandic Freshwater Radiocarbon Reservoir
- 15 Effect: Implications for lacustrine <sup>14</sup>C chronologies. The Holocene 21, 1073-1080.
- Ascough, P.L., Church, M.J., Cook, G.T., Dunbar, E., Gestsdóttir, H., McGovern, T.H.,
- Dugmore, A.J., Friðriksson, A., Edwards, K.J. 2012. Radiocarbon reservoir effects in
- 18 human bone collagen from northern Iceland. Journal of Archaeological Science 39,
- 19 2261-2271.
- 20 Ascough, P.L., Church, M.J., Cook, G.T., Einarsson, Á., McGovern, T.H., Dugmore,
- 21 A.J., Edwards, K.J. In press. Stable isotopic ( $\delta^{13}$ C and  $\delta^{15}$ N) characterization of key
- 22 faunal resources on Norse period settlements in North Iceland. Journal of the North
- 23 Atlantic.
- Bahar, B., Schmidt, O., Moloney, A.P., Scrimgeour, C.M., Begley, I.S., Monahan, F.J.
- 25 2008. Seasonal variation in the C, N and S stable isotope composition of retail organic
- and conventional Irish beef. Food Chemistry 106, 1299-1305.
- 27 Barrett, J., Johnstone, C., Harland, J., Van Neer, W., Ervynck, A., Makowiecki, D.,
- Heinrich, D., Hufthammer, A.K., Enghoff, I.B., Amundsen, C., Christiansen, J.S., Jones,
- 29 A.K.G., Locker, A., Hamilton-Dyer, S., Jonsson, L., Lõugas, L., Roberts, C., Richards,

- 1 M. 2008. Detecting the medieval cod trade: a new method and first results. Journal of
- 2 Archaeological Science 35, 850-861.
- 3 Barrett, J.H., Orton, D., Johnstone, C., Harland, J., Van Neer, W., Ervynck, A., Roberts,
- 4 C., Locker, A., Amundsen, C., Enghoff, I.B., Hamilton-Dyer, S., Heinrich, D.,
- 5 Hufthammer, A.K., Jones, A.K.G., Jonsson, L., Makowiecki, D., Pope, P., O'Connell,
- 6 T.C., de Roo, T., Richards, M. 2011. Interpreting the expansion of sea fishing in
- 7 medieval Europe using stable isotope analysis of archaeological cod bones. Journal of
- 8 Archaeological Science 38, 1516-1524.
- 9 Bjarnason, Á.H. 1991. Vegetation on lava fields in the Hekla area, Iceland. Acta
- 10 Phytogeographica Suecica 77, 3-105.
- Bogaard, A., Heaton, T.H.E., Poulton, P., Merbach, I. 2007. The impact of manuring on
- 12 nitrogen isotope ratios in cereals: archaeological implications for reconstruction of diet
- and crop management practices. Journal of Archaeological Science 34, 335-343.
- 14 Bronk Ramsey, C. 1995. Radiocarbon Calibration and Analysis of Stratigraphy: The
- 15 OxCal Program. Radiocarbon 37, 425-430.
- Bronk Ramsey, C. 2001. Development of the radiocarbon program OxCal. Radiocarbon
- 17 43, 355-363.
- 18 Claypool, G.E., Holser, W.T., Kaplan, I.R., Sakai, H., Zak, I. 1980. The age curves of
- 19 sulphur and oxygen isotopes in marine sulphate and their mutual interpretation. Chemical
- 20 Geology 28, 199-260.
- 21 Craig, O.E., Ross, R., Andersen, S.H., Milner, N., Bailey, G.N. 2006. Focus: sulphur
- 22 isotope variation in archaeological marine fauna from northern Europe. Journal of
- 23 Archaeological Science 33, 1642-1646.
- 24 Craig, O.E., Biazzo, M., Colonese, A.C., Di Giuseppe, Z., Martinez-Labarga, C., Lo
- 25 Vetro, D., Lelli, R., Martini, F., Rickards, O. 2010. Stable isotope analysis of Late Upper
- 26 Palaeolithic human and faunal remains from Grotta del Romito (Cosenza), Italy. Journal
- of Archaeological Science 37, 2504-2512.
- 28 DeNiro, M.J., Epstein, S. 1978. Influence of diet on the distribution of carbon isotopes
- 29 in animals. Geochimica et Cosmochimica Acta 42, 495-506.

- 1 DeNiro, M.J., Epstein, S. 1981. Influence of diet on the distribution of nitrogen isotopes
- 2 in animals. Geochimica et Cosmochimica Acta 45, 341-351.
- 3 DeNiro, M.J. 1985. Postmortem preservation and alteration of in *vivo* bone collagen
- 4 isotope ratios in relation to palaeodietary reconstruction. Nature 317, 806-809.
- 5 Dufour, E., Bocherens, H., Mariotti, A. 1999. Palaeodietary Implications of Isotopic
- 6 Variability in Eurasian Lacustrine Fish. Journal of Archaeological Science 26, 617-627.
- 7 Dugmore, A.J., Church, M.J., Buckland, P.C., Edwards, K.J., Lawson, I., McGovern,
- 8 T.H., Panagiotakopulu, E., Simpson, I.A., Skidmore, P., Sveinbjarnardóttir, G. 2005.
- 9 The Norse *landnám* on the north Atlantic islands: an environmental impact assessment.
- 10 Polar Record 41, 21-37.
- Dugmore, A.J., McGovern, T.H., Vésteinsson, O., Arneborg, J., Streeter, R., Keller, C.
- 12 2012. Cultural adaptation, compounding vulnerabilities and conjunctures in Norse
- 13 Greenland. Proceedings of the National Academy of Sciences 109, 3658-3663.
- Edwald, Á., McGovern, T.H. 2009. Skútustaðir Midden Investigations Mývatn Northern
- 15 Iceland 2008. . (Available for download from:
- 16 http://www.nabohome.org/uploads/tommcgovern/Skutustadir\_Field\_Report\_for\_NABO\_
- website.pdf).
- Edwald, Á., McGovern, T.H. 2010. Skútustaðir Midden Investigations Mývatn Northern
- 19 Iceland 2009. (Available for download from:
- 20 http://www.nabohome.org/publications/fieldreports/SkutustadirField%20Report20092251
- 21 0.pdf).
- 22 Einarsson, Á. 2004. Lake Myvatn and the River Laxá: An introduction. Aquatic
- 23 Ecology 38, 111-114.
- 24 Einarsson, Á., Aldred, O. 2011. The archaeological landscape of northeast Iceland: a
- 25 ghost of a Viking Age society. In: Remote Sensing for Archaeological Heritage
- 26 Management. EAC (Europae Archaeologiae Consilium) Publications. Pp. 243-258.
- 27 Einarsson, M. Á. 1979. Climatic conditions of the Lake Mývatn area. Oikos 32, 29-37.

- 1 Erskine, P.D., Bergstrom, D.M., Schmidt, S., Stewart, G.R., Tweedie, C.E., Shaw, J.D.
- 2 1998. Subantarctic Macquarie Island a model ecosystem for studying animal-derived
- 3 nitrogen sources using <sup>15</sup>N natural abundance. Oecologia 117, 187-193.
- 4 Faure, G. 1977. Sulfur. In: *Principles of Isotope Geology*. Wiley: New York, 523-552.
- 5 Fraser, R.A., Bogaard, A., Heaton, T., Charles, M., Jones, G., Christensen, B.T.,
- 6 Halstead, P., Merbach, I., Poulton, P.R., Sparkes, D., Styring, A.K. 2011. Manuring and
- stable nitrogen isotope ratios in cereals and pulses: towards a new archaeobotanical
- 8 approach to the inference of land use and dietary practices. Journal of Archaeological
- 9 Science 38, 2790-2804.
- 10 Fritzsche, F., Tichomirowa, M. 2006. Signal improvement in elemental
- analyzer/continuous flow isotope ratio mass spectrometry for samples with low sulfur
- contents using a pre-concentration technique for on-line concentration adjustment. Rapid
- 13 Communications in Mass Spectrometry 20, 1679-1682.
- 14 Fry, B., Garritt, R., Tholke, K., Neill, C., Michener, R.H., Mersch, F.J., Brand, W. 1996.
- 15 Cryoflow: Cryofocussing Nanomole Amounts of CO<sub>2</sub>, N<sub>2</sub>, and SO<sub>2</sub> from an Elemental
- Analyzer for Stable Isotope Analysis. Rapid Communications in Mass Spectrometry 10,
- 17 953-958.
- 18 Fry, B. 2007. Coupled N, C and S stable isotope measurements using a dual-column gas
- 19 chromatography system. Rapid Communications in Mass Spectrometry 21, 750-756.
- 20 Gardarsson, A. 2006. Temporal processes and duck populations: examples from
- 21 Mývatn. Hydrobiologia 567, 89-100.
- 22 Gíslason, S.R., Snorrason, Á., Kristmannsdóttir, H.K., Sveinbjörnsdóttir, Á.E.,
- Torssander, P., Ólafsson, J., Castet, S. Dupré, B. 2002. Effects of volcanic eruptions on
- 24 the CO<sub>2</sub> content of the atmosphere and the ocean: the 1996 eruption and flood within the
- 25 Vatnajökull Glacier, Iceland. Chemical Geology 190, 181-205.
- 26 Gíslason, S.R., Torssander, P. 2006. Response of Sulfate Concentration and Isotope
- 27 Composition in Icelandic Rivers to the Decline in Global Atmospheric SO<sub>2</sub> Emissions
- into the North Atlantic Region. Environmental Science & Technology 40, 680-686.

- 1 Giesemann, A., Jäger, H.-J., Norman, A.L., Krouse, H.R., Brand, W.A. 1994. On-line
- 2 Sulfur-Isotope Determination Using Elemental Analyzer Coupled to a Mass
- 3 Spectrometer. Analytical Chemistry 66, 2816-2819.
- 4 Grassineau, N.V., Mattey, D.P., Lowry, D. 2001. Sulfur Isotope Analysis of Sulfide and
- 5 Sulfate Minerals by Continuous Flow-Isotope Ratio Mass Spectrometry. Analytical
- 6 Chemistry 73, 220-225.
- 7 Grassineau, N.V. 2006. High-precision EA-IRMS analysis of S and C isotopes in
- 8 geological materials. Applied Geochemistry 21, 756-765.
- 9 Gratton, C., Donaldson, J., Vander Zanden, M.J. 2008. Ecosystems Linkages Between
- 10 Lakes and the Surrounding Terrestrial Landscape in Northeast Iceland. Ecosystems 11,
- 11 764-774.
- Hallsdóttir, M. 1987. Pollen Analytical Studies of Human Influence on Vegetation in
- 13 Relation to the Landnám Tephra Layer in Southwest Iceland. Lundqua Thesis 18,
- 14 Department of Quaternary Geology, Lund University.
- Hansen, T., Burmeister, A., Sommer, U. 2009. Simultaneous  $\delta^{15}$ N,  $\delta^{13}$ C and  $\delta^{34}$ S
- 16 measurements of low-biomass samples using a technically advanced high sensitivity
- elemental analyzer connected to an isotope ratio mass spectrometer. Rapid
- 18 Communications in Mass Spectrometry 23, 3387-3393.
- 19 Harris, E. Sinha, B., Hoppe, P., Crowley, J.N., Ono, S., Foley, S. 2012. Sulfur isotope
- 20 fractionation during oxidation of sulfur dioxide: gas-phase oxidation by OH radicals and
- 21 aqueous oxidation by H<sub>2</sub>O<sub>2</sub>, O<sub>3</sub> and iron catalysis. Atmospheric Chemistry and Physics
- 22 12, 407-424.
- 23 Hedges, R.E.M., Clement, J.G., Thomas, C.D.L., O'Connell, T.C. 2007. Collagen
- turnover in the adult femoral mid-shaft: Modeled from anthropogenic radiocarbon tracer
- 25 measurements. American Journal of Physical Anthropology 133, 808–816.
- Hersteinsson, P., MacDonald, D.W. 1996. Diet of arctic foxes (Alopex lagopus) in
- 27 Iceland. Journal of Zoology 240, 457-474.

- 1 Hicks, M.T., McGovern, T.H., Harrison, R. 2009. A Preliminary Report of the 2008
- 2 Midden Excavation at Skútustaðir, N Iceland. (Available for download from:
- 3 <u>http://www.nabohome.org/uploads/fsi/FS419-08271\_SKU08.pdf</u>).
- 4 Hicks, M.T. 2010. Skútustaðir: An Interim Zooarchaeological Report following the 2009
- 5 Field Season. (Available for download from:
- 6 <a href="http://www.nabohome.org/publications/labreports/NORSEC\_SKU\_InterimZooachaeologi">http://www.nabohome.org/publications/labreports/NORSEC\_SKU\_InterimZooachaeologi</a>
- 7 calReport2009\_5\_5\_2010.pdf).
- 8 Hicks, M.T., Pálsdóttir, L.B. 2011. Excavations at Skútustaðir, Mývatn Northern
- 9 Iceland: Preliminary Field Report After the Excavation Season June July 2010.
- 10 (Available for download from: http:// http://www.nabohome.org/uploads/fsi/FS457-
- 11 08273Skutustadir\_FieldReport.pdf)
- Hildebrand, L.W.; Torssander, P. 1998. Sulfur isotope ratios from the Katla Volcanic
- 13 Centre With implications for mantle heterogeneities? In *Water-Rock Interaction*;
- 14 Arehart, G.B., Hulston, J.R. Eds.; Balkema: Rotterdam; 451-454.
- Hobson, K.A., Clark, R.G. 1992. Assessing Avian Diets Using Stable Isotopes I:
- 16 Turnover of <sup>13</sup>C in Tissues. The Condor 94, 181-188.
- Holm, N.G., Gíslason, S.R., Sturkell E., Torssander, P. 2010. Hekla cold springs
- 18 (Iceland): groundwater mixing with magmatic gases. Isotopes in Environmental and
- 19 Health Studies 46, 180-189.
- 20 Howcroft, R., Eriksson, G., Lidén, K. 2012. Conformity in Diversity? Isotopic
- 21 Investigations of Infant Feeding Practices in Two Iron Age Populations From Southern
- Öland, Sweden. American Journal of Physical Anthropology 149, 217-230.
- Hu, Y., Shang, H., Tong, H., Nehlich, O., Liu, W., Zhao, C., Yu, J., Wang, C., Trinkaus,
- 24 E. Richards, M.P. 2009. Stable isotope dietary analysis of the Tianyuan 1 early modern
- 25 human. Proceedings of the National Academy of Sciences 106, 10971-10974.
- Jakobsson, S.P., Jónasson, K., Sigurdsson, I.A. 2008. The three igneous rock series of
- 27 Iceland. Jökull 58, 117-138.

- 1 Keaveney, E.M., Reimer, P.J. 2012. Understanding the variability in freshwater
- 2 radiocarbon reservoir offsets: a cautionary tale. Journal of Archaeological Science 39,
- 3 1306-1316.
- 4 Kristmannsdóttir, H., Ármannsson, H. 2004. Groundwater in the Lake Myvatn area,
- 5 northern Iceland: Chemistry, origin and interaction. Aquatic Ecology 38, 115-128.
- 6 Krouse, H.R. 1977. Sulphur isotope abundance elucidate uptake of atmospheric sulphur
- 7 emission by vegetation. Nature 65, 45-46.
- 8 Lamb, A.L., Melikian, M., Ives. R., Evans, J. 2012. Multi-isotope analysis of the
- 9 population of the lost medieval village of Auldhame, East Lothian, Scotland. Journal of
- 10 Analytical Atomic Spectrometry 27, 765-777.
- Lawson, I.T., Gathorne-Hardy, F.J., Church, M.J., Newton, A.J., Edwards, K.J.,
- Dugmore, A.J., Einarsson, Á. 2007. Environmental impacts of the Norse settlement:
- palaeoenvironmental data from Mývatnssveit, northern Iceland. Boreas 36, 1-19.
- Longin, R. 1971. New Method Of Collagen Extraction For Radiocarbon Dating. Nature
- 15 230, 241-242.
- Macko, S.A., Engel, M.H., Andrusevich, V., Lubec, G. O'Connell, T.C., Hedges, R.E.M.
- 17 1999. Documenting the diet in ancient human populations through stable isotope analysis
- of hair. Philosophical Transactions of the Royal Society B 354, 65-76.
- 19 Mateo, M.A., Serrano, O., Serrano, L., Michener, R.H. 2008. Effects of sample
- 20 preparation on stable isotopes ratios of carbon and nitrogen in marine invertebrates:
- 21 implications for food web studies using stable isotopes. Oecologia 157, 105-115.
- 22 McGovern, T.H., Perdikaris, S., Einarsson, A., Sidell, J. 2006. Coastal connections,
- 23 local fishing, and sustainable egg harvesting: patterns of Viking Age inland wild resource
- 24 use in Mývatn district, northern Iceland. Environmental Archaeology 11, 187-205.
- 25 McGovern, T.H., Vésteinsson, O., Fridriksson, A., Church, M., Lawson, I., Simpson,
- 26 I.A., Einarsson, A., Dugmore, A., Cook, G., Perdikaris, S., Edwards, K.J., Thomson,
- 27 A.M., Adderley, W.P., Newton, A., Lucas, G., Edvardsson, R., Aldred, O., Dunbar, E.
- 28 2007. Landscapes of settlement in Northern Iceland: Historical ecology of human impact
- and climate fluctuation on the millennial scale. American Anthropologist 109, 27-51.

- 1 McKee, K.L., Feller, I.C., Popp, M., Wanek, W. 2002. Mangrove Isotopic (δ<sup>15</sup>N and
- $\delta^{13}$ C) fractionation across a nitrogen vs. phosphorus limitation gradient. Ecology 83,
- 3 1065-1075.
- 4 Nakai, N., Jensen, M.L. 1964. The kinetic isotope effect in the bacterial reduction and
- 5 oxidation of sulfur. Geochimica et Cosmochimica Acta 28, 1893-1912.
- 6 Nehlich, O., Richards, M.P. 2009. Establishing quality criteria for sulphur isotope
- 7 analysis of archaeological bone collagen. Archaeological and Anthropological Sciences
- 8 1, 59-75.
- 9 Nehlich, O. Borić, D., Stefanović, S., Richards, M.P. 2010. Sulphur isotope evidence for
- 10 freshwater fish consumption: a case study from the Danube Gorges, SE Europe. Journal
- of Archaeological Science 37, 1131-1139.
- Nehlich, O., Fuller, B.T., Jay, M., Mora, A., Nicholson, R.A., Smith, C.I., Richards, M.T.
- 2011. Application of sulphur isotope ratios to examine weaning patterns and freshwater
- 14 fish consumption in Roman Oxfordshire, UK. Geochimica et Cosmochimica Acta 75,
- 15 4963-4977.
- Nehlich, O., Fuller, B.T., Márquez-Grant, N., Richards, M.P. 2012. Investigation of
- 17 Diachronic Dietary Patterns on the Islands of Ibiza and Formentera, Spain: Evidence from
- 18 Sulfur Stable Isotope Ratio Analysis. American Journal of Physical Anthropology 149,
- 19 115-124.
- 20 Newton, R., Bottrell, S. 2007. Stable isotopes of carbon and sulphur as indicators of
- environmental change: past and present. Journal of the Geological Society 164, 691-708.
- Nriagu, J.O., Coker, R.D. 1978. Isotopic composition of sulphur in atmospheric
- precipitation around Sudbury, Ontario. Nature 274, 883-885.
- 24 Oelze, V.M., Nehlich, O., Richards, M.P. 2012a. 'There's No Place Like Home' No
- 25 Isotopic Evidence for Mobility at the Early Bronze Age Cemetery of Singen, Germany.
- 26 Archaeometry 54, 752-778.
- 27 Oelze, V.M., Koch, J.K., Kupke K., Nehlich, O., Zäuner, S., Wahl, J., Weise, S.M.,
- 28 Rieckhoff, S., Richards, M.P. 2012b. Multi-Isotopic Analysis Reveals Individual

- 1 Mobility and Diet at the Early Iron Age Monumental Tumulus of Magdalenenberg,
- 2 Germany. American Journal of Physical Anthropology 148, 406-421.
- 3 Osorio, M.T., Moloney, A.P., Schmidt, O., Monahan, F.J. 2011. Beef Authentication
- 4 and Retrospective Dietary Verification Using Stable Isotope Ratio Analysis of Bovine
- 5 Muscle and Tail Hair. Journal of Agricultural and Food Chemistry 59, 3295-3305.
- 6 Peterson, B.J., Fry, B. 1987. Stable isotopes in ecosystem studies. Annual Review of
- 7 Ecological Systems 18, 293-320.
- 8 Peterson, B.J., Howarth, R.W., Garritt, R.H. 1985. Multiple stable isotopes used to trace
- 9 the flow of organic matter in estuarine food webs. Science 227, 1361-1363.
- 10 Privat, K.L., O'Connell, T.C., Hedges, R.E.M. 2007. The distinction between
- 11 freshwater- and terrestrial-based diets: methodological concerns and archaeological
- applications of sulphur stable isotope analysis. Journal of Archaeological Science 34,
- 13 1197-1204.
- 14 Rees, C.E., Jenkins, W.J, Monster, J. 1978. The sulphur isotopic composition of ocean
- water sulphate. Geochimica et Cosmochimica Acta 42, 377-381.
- Reimer, P.J., Baillie, M.G.L., Bard, E., Bayliss, A., Beck, J.W., Blackwell, P.G., Bronk
- 17 Ramsey, C., Buck, C.E., Burr, G.S., Edwards, R.L., Friedrich, M., Grootes, P.M.,
- 18 Guilderson, T.P., Hajdas, I., Heaton, T.J., Hogg, A.G., Hughen, K.A., Kaiser, K.F.,
- 19 Kromer, B., McCormac, F.G., Manning, S.W., Reimer, R.W., Richards, D.A., Southon,
- J.R., Talamo, S., Turney, C.S.M., van der Plicht, J., Weyhenmeyer, C.E. 2009. INTCAL
- 21 09 and MARINE09 radiocarbon age calibration curves, 0-50,000 years Cal BP.
- 22 Radiocarbon 51, 1111-1150.
- 23 Richards, M.P., Fuller, B.T., Hedges, R.E.M. 2001. Sulphur isotopic variation in ancient
- bone collagen from Europe: implications for human palaeodiet, residence mobility, and
- 25 modern pollutant studies. Earth and Planetary Science Letters 191, 185-190.
- 26 Richards, M.P., Fuller, B.T., Sponheimer, M., Robinson, T. Ayliffe, L. 2003. Sulphur
- 27 Isotopes in Palaeodietary Studies: a Review and Results from a Controlled Feeding
- 28 Experiment. International Journal of Osteoarchaeology 13, 37-45.

- 1 Robinson, Z.P., Fairchild, I.J., Spiro, B. 2009. The sulphur isotope and hydrochemical
- 2 characteristics of Skeiðarársandur, Iceland: identification of solute sources and
- 3 implications for weather processes. Hydrological Processes 23, 2212-2224.
- 4 Rummel, S., Hoelzl, S., Horn, P., Rossmann, A., Schlicht, C. 2010. The combination of
- 5 stable isotope abundance ratios of H, C, N and S with <sup>87</sup>Sr/<sup>86</sup>Sr for geographical origin
- 6 assignment of orange juices. Food Chemistry 118, 890-900.
- 7 Russell, N., Cook, G.T., Ascough, P., Barrett, J.H., Dugmore, A. 2011. Species specific
- 8 marine radiocarbon reservoir effect: a comparison of  $\Delta R$  values between *Patella vulgata*
- 9 (limpet) shell carbonate and *Gadus morhua* (Atlantic cod) bone collagen. Journal of
- 10 Archaeological Science 38, 1008-1015.
- 11 Schoeninger, M.J., DeNiro, M.J., Tauber, H. 1983. Stable Nitrogen Isotope Ratios of
- 12 Bone Collagen Reflect Marine and Terrestrial Components of Prehistoric Human Diet.
- 13 Science 220, 1381-1383.
- 14 Schoeninger, M.J., DeNiro, M.J. 1984. Nitrogen and carbon isotopic composition of
- bone collagen from marine and terrestrial animals. Geochimica et Cosmochimica Acta
- 16 48, 625-639.
- 17 Skrzypek, G., Kałużny, A., Wojtuń, B., Jędrysek, M.O. 2007. The carbon stable isotopic
- 18 composition of mosses: A record of temperature variations. Organic Geochemistry 38,
- 19 1770-1781.
- 20 Skrzypek, G., Paul, D., Wojtuń, B. 2008. Stable isotope composition of plants and peat
- 21 from Arctic mire and geothermal area in Iceland. Polish Polar Research 29, 365-376.
- 22 Slota, P.J., Jull, A.J.T., Linick, T.W., Toolin, L.J. 1987. Preparation of small samples for
- <sup>14</sup>C accelerator targets by catalytic reduction of CO. Radiocarbon 29, 303-306.
- 24 Strauss, H. 1997. The isotopic composition of sedimentary sulfur through time.
- 25 Palaeogeography, Palaeoclimatology, Palaeoecology 32, 97-118.
- Tanz, N., Schmidt, H-L. 2010.  $\delta^{34}$ S-Value Measurements in Food Origin Assignments
- 27 and Sulfur Isotope Fractionations in Plants and Animals. Journal of Agricultural and
- 28 Food Chemistry 58, 3139-3146.

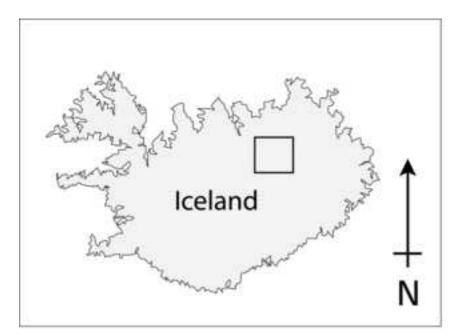
- 1 Tauber, H. 1981. <sup>13</sup>C evidence for dietary habits of prehistoric man in Denmark. Nature
- 2 292, 332-333.
- 3 Taylor, B.E., Wheeler, M.C. 1984. Isotopic composition of sulphate in acid mine
- 4 drainage as measure of bacterial oxidation. Nature 308, 538-541.
- 5 Torssander, P. 1988. Sulfur Isotope Ratios of Icelandic Lava Incrustations and Volcanic
- 6 Gases. Journal of Volcanology and Geothermal Research 35, 227-235.
- 7 Torssander, P. 1989. Sulfur isotope ratios of Icelandic rocks. Contributions to
- 8 Mineralogy and Petrology 102, 18-23.
- 9 Trust, B.A., Fry, B. 1992. Stable sulphur isotopes in plants: a review. Plant, Cell and
- 10 Environment 15, 1105-1110.
- 11 Van der Merwe, N.J., Vogel, J.C. 1978. <sup>13</sup>C Content of human collagen as a measure of
- prehistoric diet in woodland North America. Nature 276, 815-816.
- 13 Vandeputte, K., Moens, L., Dams, R. 1996. Improved sealed-tube combustion of organic
- samples to CO<sub>2</sub> for stable isotopic analysis, radiocarbon dating and percent carbon
- determinations. Analytical Letters 29, 2761-73.
- 16 Vésteinsson, O. 1998. Patterns of settlement in Iceland: A study in prehistory. Saga
- 17 Book 28, 1–29.
- 18 Vésteinsson, O., McGovern, T.H., Keller, C. 2002. Enduring Impacts: Social and
- 19 Environmental Aspects of Viking Age Settlement in Iceland and Greenland.
- 20 Archaeologia Islandica 2, 98-136.
- Vika, E. 2009. Strangers in the grave? Investigating local provenance in a Greek Bronze
- Age mass burial using  $\delta^{34}$ S analysis. Journal of Archaeological Science 36, 2024-2028.
- 23 Wadleigh, M.A., Schwarcz, H.P., Kramer, J.R. 1994. Sulphur isotope tests of seasalt
- 24 correction factors in precipitation: Nova Scotia, Canada. Water, Air and Soil Pollution
- 25 77, 1-16.
- Wadleigh, M.A. 2003. Lichens and atmospheric sulphur: what stable isotopes reveal.
- 27 Environmental Pollution 126, 345-351.

- 1 Wang, Y.M., Wooller, M.J. 2006. The stable isotopic (C and N) composition of modern
- 2 plants and lichens from northern Iceland: with ecological and paleoenvironmental
- 3 implications. Jökull 56, 27-38.
- 4 Yun, M., Mayer, B., Taylor, S.W. 2005.  $\delta^{34}$ S measurements on organic materials by
- 5 continuous flow isotope ratio mass spectrometry. Rapid Communications in Mass
- 6 Spectrometry 19, 1429-1436.

- 7 Yun, M., Wadleigh, M.A., Mayer, B. 2010. Variations of sulfur isotope ratios in a single
- 8 lichen thallus: A potential historical archive for sulfur pollution. Environmental Pollution
- 9 158, 3534-3538
- 20 Zazzo, A., Monahan, F.J., Moloney, A.P., Green, S., Schmidt, O. 2011. Sulphur isotopes
- in animal hair track distance to sea. Rapid Communications in Mass Spectrometry 25,
- 12 2371-2378.

#### Figure captions

- Figure 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.
- Figure 2:  $\delta^{34}$ S values for various Icelandic sulphur sources.
- Figure 3:  $\delta^{13}C$  and  $\delta^{15}N$  values for various flora and fauna from around Iceland and the Lake Mývatn region.
- Figure 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.
- Figure 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.
- Figure 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.



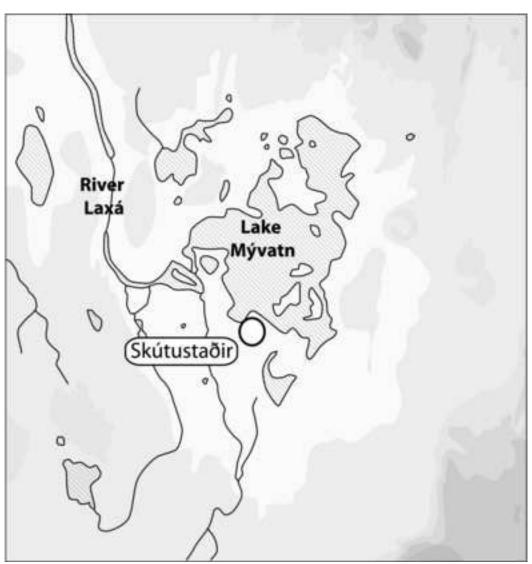
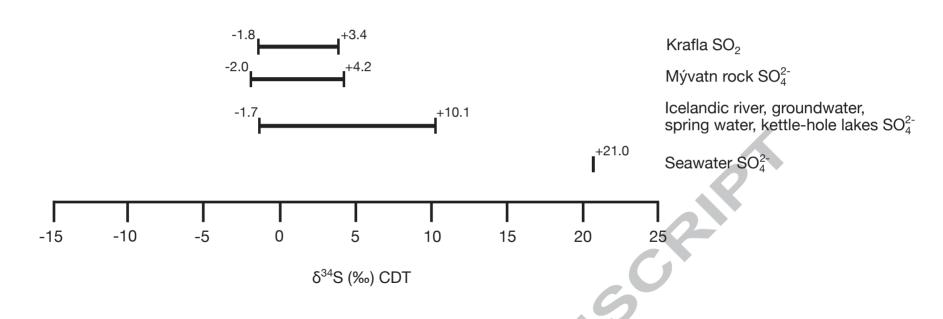
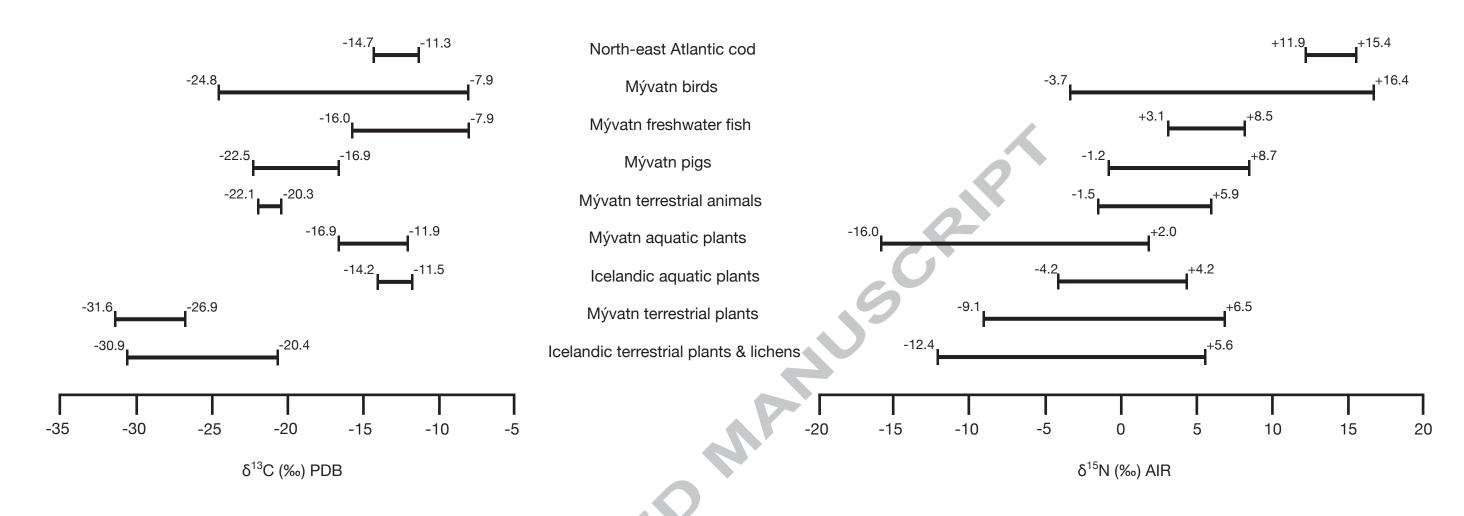
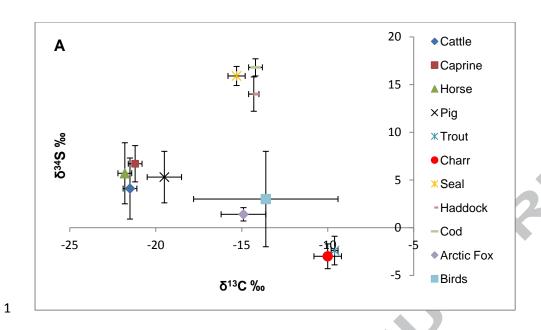
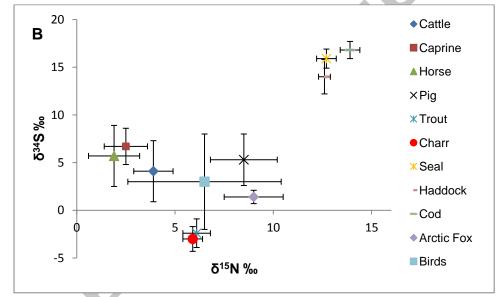


Figure 2



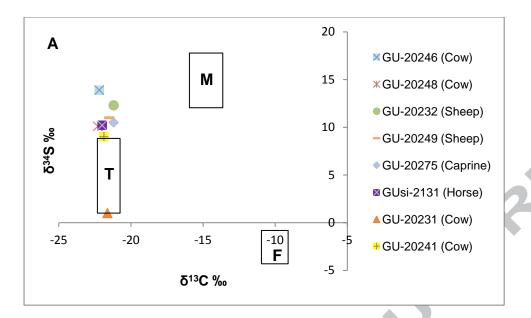


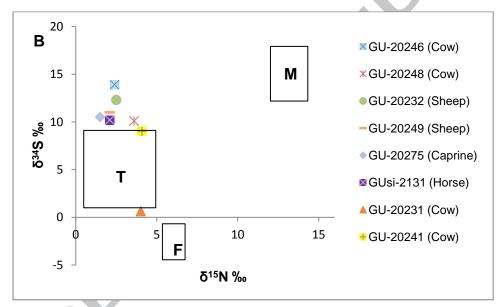




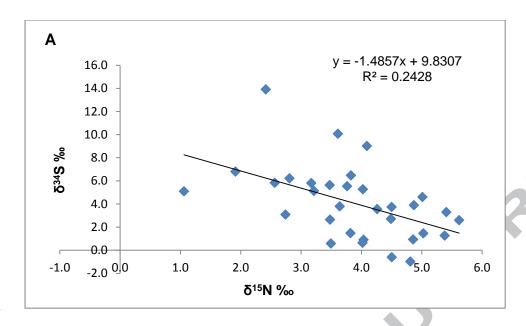
3 Figure 4

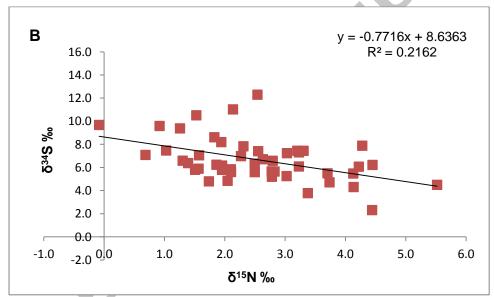
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3 Figure 5





3 Figure 6

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Species	N	δ <sup>34</sup> S [‰]	S wt%	δ <sup>13</sup> C [‰]	δ <sup>15</sup> N [‰]
Cow	32	$4.1 \pm 3.2$	$0.21 \pm 0.05$	$-21.5 \pm 0.4$	3.9 ± 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	$5.7 \pm 3.2$	$0.20\pm0.02$	$-21.8 \pm 0.4$	$1.9 \pm 1.3$
Trout	5	$-2.4 \pm 1.5$	$0.53 \pm 0.04$	$-9.6 \pm 0.2$	$6.1 \pm 0.7$
Charr	7	$-3.0 \pm 1.3$	$0.58 \pm 0.03$	$-10.0 \pm 0.8$	$5.9 \pm 0.5$
Haddock	3	$14.0\pm1.8$	$0.46 \pm 0.03$	$-14.3 \pm 0.3$	$12.6\pm0.3$
Cod	6	$16.8 \pm 0.9$	$0.50 \pm 0.02$	$-14.2 \pm 0.4$	$13.9 \pm 0.5$
Seal	6	$15.9\pm1.0$	$0.21 \pm 0.05$	$-15.3 \pm 0.5$	$12.7 \pm 0.5$
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	$1.4 \pm 0.7$	$0.25 \pm 0.03$	$-14.9 \pm 1.3$	$9.0 \pm 1.5$

Table 1: Mean and standard deviations (1 $\sigma$ ) of bone collagen  $\delta^{34}$ S,  $\delta^{13}$ C and  $\delta^{15}$ N values

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Species group	N	δ <sup>34</sup> S [‰]	δ <sup>13</sup> C [‰]	δ <sup>15</sup> 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 2: Mean and standard deviations (1σ) of terrestrial, freshwater and marine animal

6 bone collagen from Skútustaðir, Iceland.

<sup>3</sup> for animals from Skútustaðir, Iceland.

Species	N	C:S Ratio	N:S Ratio	%S	C:S Ratio	N:S Ratio	%S
		(ave.)	(ave.)	(ave.)			
Cow	32	544 ± 127	164 ± 38	$0.21 \pm 0.05$	503*	158*	0.25*
Sheep/Goat	48	$545 \pm 116$	$165 \pm 35$	$0.21 \pm 0.05$	521*	163*	0.26*
Horse	5	$575 \pm 125$	$172 \pm 41$	$0.20 \pm 0.03$	540*	166*	0.23*
Pig	3	$589 \pm 38$	$179 \pm 13$	$0.17 \pm 0.01$	401*	124*	0.32*
Char/Trout	12	$190 \pm 11$	$55 \pm 3$	$0.56 \pm 0.04$	180*	55*	0.64*
Cod/Haddock	9	$186 \pm 18$	$58 \pm 7$	$0.48 \pm 0.03$	196*	61*	0.62*
Seal	6	$579 \pm 33$	$175 \pm 11$	$0.21 \pm 0.05$	472*	148*	0.26*
Birds	11	$397 \pm 56$	$120\pm16$	$0.29 \pm 0.03$	417*	128*	0.29*
Arctic fox	3	$408 \pm 79$	$122 \pm 25$	$0.25 \pm 0.03$	N/A	N/A	N/A
All Mammals & Birds	108	$528 \pm 123$	$160 \pm 37$	$0.22 \pm 0.05$	600 ± 300*	200 ± 100*	0.15-0.35*
All Fish	21	$188 \pm 14$	$56 \pm 5$	$0.53 \pm 0.05$	$175 \pm 50*$	$60 \pm 20*$	0.4-0.8*

Table 3: Mean and standard deviations (1σ) of C:S and N:S ratios and %S of archaeological samples from Skútustaðir. \*Nehlich and Richards, 2009.