

Pica 8: Refining dietary reconstruction through amino acid $\delta^{13}\text{C}$ analysis of tendon collagen and hair keratin

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Highlights:

A method for assessing a terrestrial or marine origin for dietary intake is proposed

The new method uses $\delta^{13}\text{C}$ values of phenylalanine, valine, and leucine

Tendon collagen is a favourable substitute to bone collagen in dietary reconstructions

Tendon is an ideal tissue for characterising the final year of an individual's life

1 Abstract

2 Stable isotope analysis of archaeological human remains is routinely applied to explore dietary habits and
3 mobility patterns. The isotope information pertaining to the period prior to death may help in identifying
4 locals and non-locals, especially when investigating individuals from the same funerary context but believed
5 to have been highly mobile across the landscape.

6 Based on the variety of the funerary goods in graves and what it is believed their diets comprised, it is
7 thought that both local and non-local individuals were buried at the inland funerary site of Pica 8 (northern
8 Chile, Late Intermediate Period, ~1,050-500 BP); however, uncertainties over the dietary intakes and
9 mobility histories of these individuals still persist. The aim of this study is to refine the dietary
10 characterization of a subset of Pica 8 individuals by increasing the temporal resolution of their dietary
11 reconstructions, specifically throughout the last period of their life, and by identifying the multiple sources of
12 food in their overall diets. This is achieved by analysing the amino acid carbon isotope composition of hair
13 keratin and, for the first time, that of tendon collagen.

14 This study proposes a new method for identifying the predominant food source (terrestrial or marine) in a
15 mixed diet using phenylalanine, valine and leucine $\delta^{13}\text{C}$ values measured in collagenous tissues. Herein,
16 tendon is proven to be an ideal tissue for isotopically characterising the final year of an individual's life. Our
17 results show that individuals previously identified as non-locals, based on long-term food consumption, had
18 in reality abandoned their original dietary habits typical of distant regions many months before death, and
19 hence had presumably relocated to the locality of Pica.

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22 Keywords: Chile; tendon; collagen; amino acid; LC-IRMS; stable isotope; palaeodiet.

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31 1. Introduction

32 Stable isotope analysis is routinely applied to archaeological human remains for characterising dietary habits
33 and mobility patterns of past populations (Makarewicz and Sealy, 2015). In recent years, attention has
34 focussed on the reconstruction of the life histories of individual identities in past societies (Knapp and van
35 Dommelen, 2008). The reconstruction of an individual's life at different points over their life course is
36 achieved by isotopically analysing multiple tissues (skeletal and non-skeletal) from the same individual,
37 which have differential deposition times and/or turnover rates (Lynnerup, 2007). In particular, the analysis of
38 soft tissues, that have fast remodelling rates, gives information pertaining to the last year/months of life
39 (Lamb, 2015). Moreover, hair retains an unaltered isotope signal locked into the keratins when the tissue was
40 growing (Petzke et al., 2010). This isotope information pertaining to the period prior to death is important
41 when investigating individuals from the same funerary context but believed to have been highly mobile
42 across the landscape, or having had access to long-distance resources. Individuals identified as non-local
43 because of their diet, based on stable isotope compositions averaged over several years (from, for instance,
44 bone collagen) may, in reality, have been consuming locally accessible resources and been part of the local
45 community for a considerable period of time before death.

46 At the inland funerary site of Pica 8 in northern Chile (Late Intermediate Period, ~1,050-500 BP),
47 uncertainties over dietary intakes and mobility histories of the buried individuals still persist, especially
48 pertaining to the last period of their life. Previous archaeological and biomolecular studies (Núñez, 1984,
49 Petruzzelli et al., 2014, Santana-Sagredo et al., 2015a) have suggested that the individuals buried at Pica may
50 have had different geographical origins and/or a high degree of mobility. Núñez (1984), who first excavated
51 the cemetery, identified the presence of non-local items among the funerary inclusions in the graves (e.g.
52 pottery, textiles, bird feathers, foods). These were thought to have been imported from either the eastern
53 Andes, Altiplano, Azapa valley, or from the coast. Stable isotope analyses of bone and tooth enamel have
54 identified three major dietary related groups of people, consisting of: (1) individuals relying mainly on
55 marine resources, complemented by some maize, (2) individuals relying mainly on maize, complemented by
56 marine resources, and (3) individuals consuming predominantly C₃ terrestrial resources (Santana-Sagredo et
57 al., 2015a).

58 Based on the variety of grave goods and diets, it can be argued that the people buried at Pica 8 represent a
59 combination of local and non-local individuals who were involved in inter-regional exchange of foodstuffs
60 and exotic objects, and/or sedentary individuals who benefited from having access to a broad range of
61 resources and maintained dietary habits distinctive of their place of origin. Despite the paucity of information
62 surrounding the placement of the burials in the cemetery, there is some evidence (i.e. broad distribution of
63 funerary goods and diets) that the Pica 8 cemetery was divided into ten sectors, A to J (Núñez, 1984,
64 Santana-Sagredo et al., 2015a, Zlatar, 1984), where people with common geographic origins, socio-
65 economic status, and/or cultural traits may have been interred.

66 In light of new radiocarbon dates of paired human and camelid tissues and estimated ^{14}C offsets, Santana-
67 Sagredo and colleagues (2017) have reconsidered the original dietary interpretation (Santana-Sagredo et al.,
68 2015a) of the Pica individuals, proposing the consumption of C_4 crops fertilised with seabird guano as a
69 major cause for the high $\delta^{15}\text{N}$ values ($>20\%$) measured in bone collagen, rather than the direct consumption
70 of marine resources. Andean archaeological and ethnohistoric records (Covey, 2000, Denevan, 2001, Julien,
71 1985, Marcus et al., 1999) report that guano was traditionally mined and marine fish were procured and dried
72 by coastal communities and transported to the highlands via llama caravans. Estimating the proportion of
73 guano-fertilised maize and marine fishes in the diet of the Pica individuals is not straightforward since the
74 practice of fertilising maize with guano increases the plant $\delta^{15}\text{N}$ values to as much as 20%, but does not
75 affect $\delta^{13}\text{C}$ values (Szpak et al., 2012a, 2012b). As a result, consumption of high-trophic level marine
76 resources (fish and mammals) and guano-fertilised C_4 crops may generate similar ranges of bulk $\delta^{13}\text{C}$ and
77 $\delta^{15}\text{N}$ values in human tissues (Szpak et al., 2012b). Single amino acid carbon isotope analysis may help in
78 identifying these diverse sources (marine, terrestrial) of food macronutrients (protein, carbohydrate, lipid) by
79 comparing the $\delta^{13}\text{C}$ values of essential and non-essential amino acids, given that different metabolic
80 pathways are involved in the processes of assimilation or synthesis of the amino acids making up whole
81 proteins (Newsholme et al., 2011, Reeds, 2000), and that guano is not affecting the carbon stable isotope
82 compositions of plant tissues (Szpak et al., 2012a).

83 The aim of the present study is to refine the dietary characterization of a subset of Pica 8 individuals by
84 increasing the temporal resolution of palaeodietary reconstructions, specifically throughout the last period of
85 their life, and by identifying the different sources of food (marine and terrestrial) that likely comprised their
86 mixed dietary intakes. This is achieved by analysing the amino acid carbon isotope composition of hair
87 keratin and, for the first time, that of tendon collagen. Tendon has the potential to be an ideal substrate for
88 characterising the period leading to/close to the time of death, considering that (1) collagen is significantly
89 more abundant in tendons ($\sim 677 \pm 57$ nmol/g wet tissue) than in the dermis ($\sim 335 \pm 64$ nmol/g wt), bone
90 ($\sim 307 \pm 71$ nmol/g wt) or skeletal muscle ($\sim 59 \pm 17$ nmol/g wt) (Kjaer et al., 2005), and that (2) the rate of
91 collagen turnover in tendon (Babraj et al., 2005, Miller et al., 2005) is more rapid than in bone (Hedges et al.,
92 2007).

93

94 2. The cemetery of Pica 8

95 Pica 8 is located approximately 80 km inland, at circa 1,300 m of elevation, on the plain of the *Pampa del*
96 *Tamarugal* (northern Chile, Fig. 1) (Jayne et al., 2016). This funerary site comprises 254 burials (Núñez,
97 1984) and dates to the Late Intermediate Period ($\sim 1,050$ -500 BP), based on ceramic seriation and
98 radiocarbon dating (Núñez, 1976, Santana-Sagredo et al., 2017, Uribe et al., 2007). The Pica oasis was part
99 of a complex system of communities situated in the Tarapacá region between the Río Camiña in the north
100 and the Río Loa in the south, and covered an altitudinal transect from the coast to the Precordillera ($\sim 2,500$
101 masl) (Uribe et al., 2007).

102 [Figure 1. Map of northern Chile showing the location of the Pica 8 site.]

103 During this period, the Pica-Tarapacá oases were connected to each other by a complex network of routes
104 and campsites, which allowed long-distance trade of resources and exotic objects between the coast and the
105 highlands, via llama caravans (Briones et al., 2005, Pomeroy, 2013). In this arid region, inter-regional
106 redistribution of surpluses between ecologically different zones was crucial, especially in times of shortage
107 of staple resources (Núñez and Dillehay, 1979, Zori and Brant, 2012). Competition over the control of this
108 trade network likely generated disputes between the local elites. The fact that artefacts related to conflict
109 (found as grave goods) were not associated with violence-related bone injuries, notwithstanding that soft
110 tissue injuries may have occurred, suggests that underlying tension was sublimated in symbolic celebrations
111 of power as a means to reinforce the leadership of certain elites (Pacheco and Retamal, 2017).

112

113 3. Materials

114 The Pica 8 collection is curated at the Department of Anthropology, University of Chile, Santiago de Chile
115 (Lemp et al., 2008), and comprises approximately 150 naturally mummified individuals. Six adult
116 individuals with varying ages, four females and two males, were selected for analysis in order to include
117 bodies deposited in burial sectors that have been suspected to be linked to people with a diversity of diets,
118 geographical origins and cultural identities (Núñez, 1984, Retamal et al., 2012, Santana-Sagredo et al.,
119 2015a). A foot tendon and a bundle of scalp hair were sampled from each individual.

120

121 4. Methods

122 4.1. Tendon collagen stable isotope analysis

123 Tendon was processed following the Finucane method (2007) originally proposed for soft tissues such as
124 skin and muscle, but increasing the number of washing steps before protein denaturation. In detail, the
125 tendons were cleaned by sonication in Milli-Q water (Merck), immersed in a mixture of
126 chloroform:methanol (2:1 v/v) (Merck), sonicated for ~20 min, and soaked overnight to remove any lipid
127 content. Every 24 hours, the chloroform:methanol (2:1 v/v) was replaced and sonicated (20 min) until all
128 lipids were removed. The samples were then rinsed six times with Milli-Q water (sonicated each time for 20
129 min). A few drops (~2-3) of 0.5M HCl (Merck) were added to the samples immersed in fresh Milli-Q water
130 to obtain a pH 3 solution. Sealed tubes were placed into a heater block for 48 h at 73°C for gelatinization.
131 The supernatant was filtered with an Ezee-Filter™ (Elkay Laboratory Products), and the residue was
132 discarded. Each filtered sample was then decanted into Pyrex® glass tubes (Corning), frozen (-20 °C), and
133 then freeze-dried (48 h).

134

135 4.1.1. Bulk carbon and nitrogen stable isotope analysis

136 The lyophilised collagen was inserted into tin capsules for the analysis of carbon and nitrogen isotope
137 compositions, which was performed using a Carlo Erba CE1110 CHN-S analyser coupled to a Fisons
138 Isochrom Continuous-flow Isotope Ratio Mass Spectrometer (GV instruments). The $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ values are
139 reported relative to international standards: V-PDB and AIR, respectively. Secondary (reference) materials
140 and in-house standards (MZ1 Maize, HAR4 Sunflower, ATP12) were used to monitor analytical precision of
141 the carbon and nitrogen isotope ratio measurements that were $\pm 0.1\%$ (1σ) for both elements.

142

143 4.1.2. Single amino acid carbon isotope analysis

144 Approximately 1 mg of tendon collagen was hydrolysed under vacuum in 1 ml of amino acid-free 6M HCl
145 (Sigma-Aldrich) at 110 °C for 24 h, after Choy et al. (2010). The hydrolysates were dried in a rotary vacuum
146 concentrator and frozen until required for analysis. Prior to analysis, samples were dissolved under
147 sonication in Milli-Q water, with the addition of 10 μl of 1mmol solution of 2-aminoisobutyric acid (Sigma-
148 Aldrich) as the internal standard (I.S.); this was to obtain a sample stock of $\sim 1.8 \mu\text{g}/\mu\text{l}$. The sample stock was
149 further diluted in Milli-Q water to deliver 10.8 to 16.2 μg of protein on the LC column (10 μl partial loop
150 injection mode).

151 LC-IRMS analysis was undertaken at the La Trobe Institute for Molecular Sciences (LIMS, La Trobe
152 University, Melbourne, Australia) using an Accela 600 pump connected to a Delta V Plus Isotope Ratio
153 Mass Spectrometer via a Thermo Scientific LC Isolink (Thermo Scientific). The amino acids were separated
154 using a mixed-mode Primesep A column (2.1x250 mm, 100 Å, 5 μm , SIELC Technologies) following the
155 chromatographic method described in Mora et al. (2017), after Smith et al. (2009). Mobile phases were:
156 Phase A = 35 μl of 1:50 96% H_2SO_4 (Merck) in 1L Milli-Q water, Phase B (1L)= 1 ml 96% H_2SO_4 (Merck)
157 and 2.28 g of $\geq 98\%$ K_3PO_4 (Sigma-Aldrich) in Milli-Q water, and Phase C (1L)= 3 ml 96% H_2SO_4 (Merck)
158 in Milli-Q water. The LC gradient program was similar to that of Mora et al. (2017), but the flow rate of the
159 analytical run was decreased to 60 - 60 $\mu\text{l}/\text{min}$ (Table 1), in order to improve the baseline resolution of the
160 isoleucine, leucine, lysine, histidine and tyrosine peaks. When needed, the flow rate was increased to 80 or
161 100 $\mu\text{l}/\text{min}$ after the tyrosine peak to gain faster sample elution.

162 [Table 1. Here]

163 Samples were analysed in duplicate. Baseline separation was achieved for all the amino acids in the tendon
164 collagen, with the exception of methionine (Fig. 2). Histidine and methionine peaks were too small to
165 generate reliable $\delta^{13}\text{C}$ values. Overall, the amino acid carbon contribution measured by LC-IRMS analysis
166 corresponded to 98.4% of the carbon present in human tendon collagen (Schofield et al., 1971).

167 [Figure 2. LC-IRMS chromatogram of tendon collagen hydrolysate (from individual SE-T3).]

168

169 4.2. Bayesian stable isotope mixing model: FRUITS

170 The Bayesian mixing model FRUITS - Food Reconstruction Using Isotopic Transferred Signals (Fernandes
171 et al., 2014) has been applied to the collagen stable isotope data produced in the current and previous studies
172 (Santana-Sagredo et al., 2015a) to achieve qualitative and quantitative estimates of the nutritional intake of
173 the Pica individuals. Although other Bayesian stable isotope mixing models have comparable features to
174 those of FRUITS (Phillips et al., 2014), we have used FRUITS as it has been commonly preferred by
175 researchers for reconstructing human palaeodiets, especially from Chilean contexts (e.g. Andrade et al.,
176 2015, King et al., 2018, Mora et al., 2017, Pestle et al., 2016, Pestle et al., 2017) in recent years. Details can
177 be found in Appendix A.

178

179 4.3. Hair keratin amino acid carbon isotope analysis

180 Amino acid $\delta^{13}\text{C}$ analysis was performed on 1 cm segments of a single hair from each individual, cut
181 longitudinally along the hair fibre, from the root to the first 10 cm. Hair samples were inserted into
182 hydrolysis tubes, and left to soak in methanol for 12-24 h in order to remove organic residues. Once dried,
183 the hair segment was hydrolysed in 6 M hydrochloric acid at 110°C until the hair fibre was completely
184 dissolved. The amount of 6M HCl used for hydrolysis was increased to $\sim 0.175 \mu\text{g}/\mu\text{l}$, compared to Mora et
185 al. (2017). Hydrolysed samples were then dried overnight at 30°C in a rotary vacuum concentrator and
186 frozen until required for analysis. For LC-IRMS injection, samples were dissolved under sonication in Milli-
187 Q water, supplemented by an internal standard (2-aminoisobutyric acid), to obtain keratin hydrolysates with
188 a concentration of approximately $0.8 \mu\text{g}/\mu\text{l}$. Samples were analysed in duplicate. The LC-IRMS analysis is
189 detailed in section 4.1.2.

190

191 5. Results and Discussion

192 5.1. Elemental and molecular composition of tissues

193 5.1.1. Assessment of the preservation of tendon collagen

194 The calculated C/N atomic ratios (3.2 to 3.4, Table 2) fall within the range of 2.9-3.6, proposed by DeNiro
195 (1985) for well-preserved bone collagen. This range is likely applicable to tendon collagen as the most
196 abundant protein in both tissues is type I collagen (Kannus, 2000, Wang, 2006), and the C/N ratios measured
197 by Ambrose (1990) in bovine tendon were comparable to those measured in bone collagen. The content by
198 weight (%) of carbon ($45.8 \pm 0.7\%$) and nitrogen ($16.0 \pm 0.2\%$) of the tendons analysed herein is comparable to
199 that measured in bovine tendon collagen ($47.6 \pm 1.5\%$ C and $16.0 \pm 0.6\%$ N) by Ambrose (1990). Based on
200 these criteria, the tendon collagens submitted for analysis were well preserved.

201 [Table 2. here]

202

203 5.1.2. Assessment of the molecular preservation of tendon collagen

204 To assess the molecular preservation of tendon collagen samples, the amino acid Area All [V] values (i.e.
205 sums of the peak areas for the ion currents at m/z 44, 45, 46), generated by LC-IRMS, were converted to
206 fractions of the total (%) and compared to (1) those derived from archaeological human bone collagen (n=12,
207 excluding tyrosine) analysed under similar chromatographic conditions, and to (2) the amino acid carbon
208 weights (%) of human tendon collagen estimated from the amino acid residues published by Schofield et al.
209 (1971). The residues were firstly multiplied by the number of carbon atoms per residue to determine the
210 amino acid carbon contribution, and the fraction of the total was calculated to make the residues comparable
211 to the LC-IRMS output. Based on this comparison, it was possible to show that the tendon collagen samples
212 had preserved their amino acid composition. Amino acid profiles (mean \pm 1 σ) are shown in Fig. 3.

213 [Figure 3. Fractions of the total (%) of amino acid peak areas measured in archaeological tendon collagen
214 (this study) and bone collagen (mean \pm 1 σ), and of amino acid carbon weights (%) of human tendon collagen
215 derived from Schofield et al. (1971).]

216

217 5.1.3. Assessment of the molecular preservation of hair keratin

218 To assess the molecular preservation of the hair keratins, the amino acid peak areas were converted to
219 fractions of the total (%) and compared to (1) those of modern human hair prepared following the same
220 procedure as for the archaeological hair, but preceded by methanol:chloroform (2:1 v/v) washings to remove
221 any residue of detergents and lipids (O'Connell et al., 2001), and to (2) the amino acid carbon weights (%) of
222 human hair derived from Wolfram (2003) and Robbins and Kelly (1970). Based on this comparison, it was
223 possible to assess that the hair keratin samples had preserved their amino acid composition. Amino acid
224 profiles (mean \pm 1 σ) are shown in Fig. 4.

225 [Figure 4. Fractions of the total (%) of amino acid peak areas measured in archaeological and modern hair
226 (mean \pm 1 σ), and of amino acid carbon weights (%) measured in human hair (Robbins and Kelly, 1970,
227 Wolfram, 2003).]

228

229 5.2. Bulk stable isotope compositions and FRUITS diet estimates

230 The $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ isotope values measured in this study from tendon collagen were compared to those
231 measured by Santana-Sagredo et al. (2015a) in bone collagen from the same individuals (Table 2). Due to the
232 differential turnover rates, rib collagen isotope composition represents an average of the dietary intake over
233 the last three to five years of life (Jørkov et al., 2009), while tendon collagen reflects the food consumed
234 during the last few months of life (Babraj et al., 2005). Previous carbon and nitrogen isotope studies (Basha

235 et al., 2016, Finucane, 2007, Iacumin et al., 1998, White and Schwarcz, 1994) on bone and soft tissues
236 (tendon, muscle, skin) from archaeological human remains have reported differences in the isotope values
237 between tissues from the same individual. Multiple and concurrent factors may be responsible, including: (1)
238 changes in diet and/or in physical condition experienced by an individual at different points in his/her life
239 course (Neuberger et al., 2013, Reitsema, 2013, Warinner and Tuross, 2010), (2) different protein (and
240 amino acid) composition of the tissues (e.g. type I collagen vs. actin and myosin) (Eastoe, 1955, Schofield et
241 al., 1971), and (3) diverse fractionation processes that may be connected to the remodelling of the various
242 proteins and/or to their specific metabolism (Ambrose and Norr, 1993, Hare et al., 1991, Tieszen et al.,
243 1983). If it is assumed that different fractionation processes exist for bone and tendon collagen, connected to
244 their specific metabolism and remodelling, these effects are likely to be much smaller than the changes in
245 diet experienced by individuals at different points in their life course, which are differentially recorded in
246 tendon and bone, depending on their turnover rates. Having taken the above into account, the diet-to-
247 collagen offset for tendon collagen is assumed herein to be comparable to that of bone.

248 Given these premises, the Bayesian mixing model Food Reconstruction Using Isotopic Transferred Signals
249 (FRUITS) (Fernandes et al., 2014) was applied to the tendon collagen isotope data produced in this study and
250 to the bone collagen isotope data published by Santana-Sagredo et al. (2015a). The food groups used in the
251 mixing model include the widest array of foods that were available through trade from the Pacific coast,
252 coastal valleys, mid-altitude valleys and highlands (Aufderheide et al., 1994, Burrell et al., 2013, DeNiro
253 and Hastorf, 1985, DeNiro, 1988, Falabella et al., 2007, Finucane et al., 2006, Gil et al., 2011, Hoeinghaus et
254 al., 2011, Hückstädt et al., 2007, Szpak et al., 2013, Szpak et al., 2014, Szpak et al., 2015, Thornton et al.,
255 2011, Tieszen and Chapman, 1992, van Der Merwe et al., 1993). The edible portion of flora and fauna (e.g.
256 animal muscle, plant fruit, tuber or grains, etc.) was calculated by applying suitable offsets (depending on the
257 type of tissue and taxonomic rank) (Codron et al., 2005, Hobson and Clark, 1992, Hobson et al., 1996, Kelly,
258 2000, Mateo et al., 2008, Sealy et al., 1987, Sholto-Douglas et al., 1991, Vogel, 1978, Warinner and Tuross,
259 2010, Yoneyama and Ohtani, 1983). A correction of 1.5‰ was applied to the $\delta^{13}\text{C}$ values of modern samples
260 to account for the Suess effect (Marino and McElroy, 1991, Schloesser et al., 2009). In Fig. 5, the tendon
261 collagen isotope data produced in this study and the bone collagen isotope data published by Santana-
262 Sagredo et al. (2015a) are compared against the edible portion of South American flora and fauna.

263 [Figure 5. Plot of $\delta^{15}\text{N}$ values versus $\delta^{13}\text{C}$ values of tendon collagen (*, this study) and bone collagen (†,
264 Santana-Sagredo et al., 2015a) from Pica 8 individuals and of the edible portion of South American flora and
265 fauna.]

266 The food groups used in the FRUITS model consist of: (1) C_4 plants (cultivated and wild), (2) marine
267 animals (fish, sea lions, shellfish), (3) C_3 plants (fruits, vegetables, legumes), and (4) terrestrial animals
268 (camelids, rodents and birds) consuming either C_3 , C_4 or mixed C_3 - C_4 plants (i.e. wild and domesticated
269 animals living at different elevations). To account for the use of guano (seabird dung) in inland maize
270 cultivation, a second model was run, substituting the ‘ C_4 plants’ food group with ‘maize manured with

271 guano'. Guano was commonly imported from the coast to increase the productivity of high-altitude
272 cultivation (Ajata López, 2013). The diet estimates generated by the FRUITS models are provided in Tables
273 3-4. The proportion of animal meat in reconstructed diets changes significantly between the two models. The
274 dietary estimates of the second model (Table 4), compared to the first (Table 3), indicate a greater
275 contribution of terrestrial animal meat and a lower contribution of marine foodstuffs. In the second model
276 (Table 4), the proportion of marine resources is lower than that originally proposed by Santana-Sagredo et al.
277 (2015a).

278 [Tables 3 and 4. Here]

279 The tendons of four individuals (SD-T24, SI-T32, SI-T3, SF-T4) recorded similar bulk isotope values ($\delta^{13}\text{C} =$
280 -7.8‰ to -8.7‰ , $\delta^{15}\text{N} = +15.5\text{‰}$ to $+17.5\text{‰}$, Table 2 and Fig. 5), which, according to FRUITS estimates,
281 reflect a dietary intake based mostly on C_4 plant products (56-72% of the food intake), complemented by
282 animal meat of terrestrial (9-34%) and/or, to a lesser extent, marine (3-15%) origin (Tables 3-4). FRUITS
283 dietary estimates (Tables 3-4), based on tendon collagen isotope data ($\delta^{13}\text{C} = -18.3\text{‰}$, $\delta^{15}\text{N} = +12.2\text{‰}$, Table
284 2 and Fig. 5), indicate that the young woman SE-T3 consumed predominantly C_3 plant products (77-79%)
285 and some meat of terrestrial animals (~11%) during the final months of her life, whereas the young woman
286 SI-T74 ($\delta^{13}\text{C} = -10.8\text{‰}$, $\delta^{15}\text{N} = +25.3\text{‰}$, Table 2 and Fig. 5) had a significant caloric contribution from
287 marine proteins (71-81%, Tables 3-4).

288

289 5.3. Amino acid carbon stable isotope compositions

290 The tendon collagen amino acid (AA) $\delta^{13}\text{C}$ values are reported in Appendix B. The $\delta^{13}\text{C}$ mass balance
291 values, calculated based on tendon amino acid composition reported by Schofield et al. (1971), differ from
292 the measured bulk $\delta^{13}\text{C}$ values by $0.55 \pm 0.11\text{‰}$. The full dataset of hair keratin amino acid $\delta^{13}\text{C}$ values is
293 provided in Appendix C.

294 The study of stable carbon isotope compositions of human proteins at the amino acid level makes it possible
295 to track the various sources of the dietary macronutrients because of the different carbon isotope
296 fractionations associated with the processes of assimilation and biosynthesis of different amino acids into
297 human proteinaceous tissues. The extent of the carbon isotope fractionation between diet and bone collagen
298 has been investigated through stable isotope analysis of tissues taken from animals raised on controlled diets
299 (Copley et al., 2004, Hare et al., 1991, Howland et al., 2003, Jim et al., 2006, Webb et al., 2017). Essential
300 amino acids present minimal isotope fractionation since they are assimilated directly from dietary proteins
301 into the body tissues (Newsholme et al., 2011, Reeds, 2000). In particular, bone collagen $\delta^{13}\text{C}$ values of some
302 essential amino acids such as leucine and phenylalanine were found to closely reflect the $\delta^{13}\text{C}$ values of the
303 respective dietary amino acids, thus being useful for identifying the source of the protein component of the
304 diet (Copley et al., 2004, Howland et al., 2003, Jim et al., 2006). At the base of the food chain, amino acid
305 $\delta^{13}\text{C}$ values of C_4 plant species are enriched in the ^{13}C isotope compared with those of the C_3 plants (Fogel

306 and Tuross, 2003), and amino acid $\delta^{13}\text{C}$ values of organic matter from marine ecosystems are enriched
307 relative to freshwater-derived amino acids (Keil and Fogel, 2001). Non-essential amino acids can be
308 synthesised *de novo*, in addition to direct assimilation. This means that the human body may synthesise these
309 amino acids through metabolic pathways involving all the three macronutrients (proteins, carbohydrates,
310 lipids) (Newsholme et al., 2011, Reeds, 2000). Among non-essential amino acids, alanine is preferentially
311 synthesized by the body, rather than routed, even under high-protein intake (Fernandes et al., 2012, Jim et
312 al., 2006), making this non-essential amino acid useful for identifying the non-protein portion of the diet.

313 A number of dietary proxies for human palaeodietary reconstruction based on bone collagen amino acid $\delta^{13}\text{C}$
314 values have been proposed (e.g. Choy et al., 2010, Corr et al., 2005, Honch et al., 2012, Webb et al., 2016).
315 Arguably the greater separation of individuals belonging to four dietary groups (HMP, HFP, C4, C3, i.e.
316 consumers of predominantly high-marine proteins, high-freshwater proteins, C4 plants, or C3 plants) was
317 achieved using the dietary proxy $\Delta^{13}\text{C}_{\text{Val-Phe}}$ and thus by plotting $\delta^{13}\text{C}$ phenylalanine vs. $\delta^{13}\text{C}$ valine values
318 (Honch et al., 2012). When the bone collagen $\Delta^{13}\text{C}_{\text{Val-Phe}}$ proxy (Honch et al., 2012) is applied to our dataset,
319 the Pica individuals all fall within the range of terrestrial consumers (Fig. 6). This is difficult to reconcile
320 with the results of the FRUITS models, as the $\Delta^{13}\text{C}_{\text{Val-Phe}}$ dietary indicator appears to underestimate the
321 marine protein intake, especially in the case of SI-T74, who has been identified through the FRUITS models
322 to have a diet containing a significant marine component (41% to 51%).

323 [Figure 6. Plot of $\Delta^{13}\text{C}_{\text{Val-Phe}}$ values for tendon collagen (this study) and bone collagen (Honch et al., 2012).
324 HMP=high-marine protein consumer; HFP=high-freshwater protein consumer; C4=C4 plant consumers;
325 C3=C3 plant consumers; Peru=mixed-diet group, Peru, Huari AD 500-900.]

326 The bi-plot $\delta^{13}\text{C}$ Phe vs. $\delta^{13}\text{C}$ Val values, proposed by Honch et al. (2012) for bone collagen, appears
327 effective in tracking the source of the terrestrial resources in the diet of the Pica individuals, being C₃ for SE-
328 T3 and C₄ for SD-T24, SI-T32, SF-T4 and SI-T3 (Fig. 7). However, the identification of SI-T74 as a marine
329 resource consumer is not straightforward.

330 [Figure 7. Plot of $\delta^{13}\text{C}$ phenylalanine values vs. $\delta^{13}\text{C}$ valine values for tendon collagen (this study) and bone
331 collagen (Honch et al., 2012).]

332 Previous studies (Choy et al., 2010, Webb et al., 2015) have shown that by using the $\delta^{13}\text{C}$ values of three
333 amino acids the extent of separation of diverse dietary groups may be increased. In our opinion, by including
334 a third essential amino acid, leucine, to the previous model of Honch et al. (2012) based on valine and
335 phenylalanine $\delta^{13}\text{C}$ values, the identification of the predominant food source can be facilitated even in a
336 mixed marine-terrestrial diet. This is because the source of carbon in the essential (bodily) amino acids
337 phenylalanine and leucine is that of dietary proteins, even under conditions of high-lipid and low-protein
338 diets. As a result of direct assimilation, $\delta^{13}\text{C}$ values of phenylalanine and leucine in proteinaceous tissues
339 reflect those of the protein component of the diet with minimal isotopic fractionation (Howland et al., 2003,
340 Newsome et al., 2011, Newsome et al., 2014). This is not true for the essential amino acid valine. Controlled

341 feeding experiments on animals (Newsome et al., 2011, Newsome et al., 2014) have shown that carbon in
342 valine may be sourced from the non-protein portion of the diet under conditions of low protein intake. It is
343 suggested that gut microflora may synthesize *de novo* essential amino acids such as valine sourcing carbon
344 from dietary carbohydrates and/or lipids (Newsome et al., 2011, Newsome et al., 2014). This implies that the
345 $\delta^{13}\text{C}$ valine value of proteinaceous tissues may be more similar to the $\delta^{13}\text{C}$ value of the bulk diet rather than
346 that of dietary proteins (Newsome et al., 2011), or be strongly correlated with whole diet $\delta^{13}\text{C}$ value
347 (McMahon et al., 2010), or have a non-significant correlation with dietary amino acids (Howland et al.,
348 2003), depending on the chosen diet. As a result of the possible processes of assimilation and/or synthesis of
349 valine from diet to body tissues, connected to a variety of dietary types, we might expect a wide range of
350 valine $\delta^{13}\text{C}$ values. Given these premises, we hypothesise that by using these three essential amino acids
351 ($\delta^{13}\text{C}$ phenylalanine, $\delta^{13}\text{C}$ valine, $\delta^{13}\text{C}$ leucine), the reconstruction of palaeodiets of individuals having a
352 mixed dietary intake will be more straightforward. The $\delta^{13}\text{C}$ values of phenylalanine and leucine may be
353 useful in tracking and thus separating different dietary groups based on the protein component of their diet,
354 and valine $\delta^{13}\text{C}$ values might be useful in tracking the non-protein portion of diet (carbohydrates and/or
355 lipids), especially under the condition of protein deficiency.

356 [Figure 8. Proposed model for assessing the origin of the predominant dietary intake (terrestrial vs. marine),
357 based on tendon collagen amino acid $\delta^{13}\text{C}$ values.]

358 As shown in Fig. 8, the $\delta^{13}\text{C}$ phenylalanine values are more negative than the leucine values for consumers
359 of mostly marine resources (e.g. SI-T74). Conversely, the leucine $\delta^{13}\text{C}$ values are more negative than the
360 phenylalanine values for consumers of mostly terrestrial resources (e.g. SD-T24, SI-T32, SE-T3). The range
361 of these essential amino acid $\delta^{13}\text{C}$ values is significantly more negative for C_3 terrestrial resource consumers
362 (e.g. SE-T3) than for the C_4 terrestrial resource consumers (e.g. SI-T32), reflecting the differential amino
363 acid $\delta^{13}\text{C}$ values of the two plant groups (Fogel and Tuross, 2003). A similar pattern was highlighted by
364 Honch et al. (2012) but for collagen $\delta^{13}\text{C}$ phenylalanine and valine values. However, valine presents more
365 variable $\delta^{13}\text{C}$ values (at least in our dataset), also likely reflecting the effect of the non-protein component of
366 the diet.

367 Unfortunately, the most extensive and complete dataset of human bone collagen $\delta^{13}\text{C}$ values published so far
368 (Honch et al., 2012) does not include leucine $\delta^{13}\text{C}$ values, making it impossible to test our hypothesis further.
369 The applicability of this Phe/Val/Leu method to various human proteinaceous tissues will need to be tested
370 in future studies.

371

372 5.4. Dietary reconstruction of selected Pica 8 individuals

373 The following interpretation is based on the assumption that, even though a wide spectrum of exotic
374 resources was available at Pica (Núñez, 1984, Uribe, 2006), it is unlikely that local individuals consumed
375 imported foods exclusively and in a consistent manner. Consumers of exotic foodstuffs (e.g. dried fish)

376 would be most likely to show a dietary intake made up of local and non-local products, with the proportion
377 of the latter varying through time depending on the nature and outcome of the commerce. Based on this
378 premise, the mobility and migratory histories of the individuals analysed are discussed, although it should be
379 noted that these interpretations are speculative, given the possible mobility of exotic foodstuffs.

380 Altogether, tendon collagen amino acid $\delta^{13}\text{C}$ values and FRUITS diet estimates (Fig. 9), based on tendon
381 collagen isotope data ($\delta^{13}\text{C} = -7.8\text{‰}$ to -8.7‰ , $\delta^{15}\text{N} = +15.5\text{‰}$ to $+17.5\text{‰}$), concur in identifying that four
382 individuals (SD-T24, SI-T32, SI-T3, SF-T4) of both sexes and different ages were consuming a terrestrial-
383 based diet made of mostly C_4 crops (56-72% of the food intake) and animal meat (9-34%), despite being
384 buried in different sectors (D, F, I) of the Pica 8 cemetery. According to the ecological zone in which the site
385 is geographically located (inland, at mid-altitude, in an arid environment), it may be assumed that this was
386 the 'local' diet consumed at Pica or in nearby communities. This is only partially in line with what has been
387 discussed in previous dietary reconstructions (Santana-Sagredo et al., 2015a, Santana-Sagredo et al., 2015b)
388 undertaken on human remains from the Chilean inland oases of Pica and Quillagua (Late Intermediate
389 Period). Both previous studies have proposed a 'local' dietary intake made up of mixed terrestrial resources,
390 being predominantly composed of maize, and marine foodstuffs. Archaeo-botanical and -faunal remains
391 recovered from Pica-Tarapacá sites of the Late Intermediate Period include C_4 crops such as maize (*Zea*
392 *mays*), rodents (*Chinchilla* sp., *Cavia porcellus*, *Lagidium viscacia*), and camelids. The marine resources had
393 to be imported from the Pacific coast and included fish (*Trachurus symmetricus*, *Cilus gilberti*), seabirds,
394 marine mammals, molluscs and crustaceans (*Mulinia* sp., *Oliva peruviana*, *Choromytilus chorus*, *Argopecten*
395 *purpuratus*) (García and Uribe, 2012, Uribe, 2006).

396 Although C_3 cultigens (e.g. beans, potato, squashes, quinoa, pepper) were cultivated and C_3 fruits were
397 gathered (*Prosopis* spp.) in Pica-Tarapacá communities of the Late Intermediate Period (García and Uribe,
398 2012), these plant products were only consumed by a small number of individuals buried at Pica (4 out of
399 35) (Santana-Sagredo et al., 2015a, Santana-Sagredo et al., 2017), at least based on the skeletons investigated
400 so far. This is surprising as the legume tree *Prosopis* spp. is a nitrogen-fixing plant that grows well in the arid
401 climate and saline soils of the *Pampa del Tamarugal*, being able to rely solely on groundwater (Fritz et al.,
402 1981, Mooney et al., 1980). Based on the paucity of consumption of C_3 plants at Pica 8, it could be implied
403 that these cultigens were less frequently cultivated in this oasis (being more easily cultivated at higher
404 altitude in the Precordillera and Altiplano, where conditions were wetter and colder), or that they were
405 accessed only by a selected group of people within the community, or alternatively that their cultivation was
406 for the purpose of trade (or any of these factors to a greater or lesser extent).

407 [Figure 9. For each individual, from top to bottom: Calorie contribution of each food group to the diet,
408 estimated via Bayesian stable isotope mixing model FRUITS, based on bulk $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ values of tendon
409 collagen (this study) and bone collagen (Santana-Sagredo et al., 2015a). (Asterisk indicates FRUITS model
410 with 'maize fertilised with guano' replacing 'C₄ plants' food group); Amino acid $\delta^{13}\text{C}$ values and calculated

411 $\delta^{13}\text{C}$ mass balance (MB) values for tendon collagen; Keratin $\delta^{13}\text{C}$ values of non-essential and essential amino
412 acids, and calculated $\delta^{13}\text{C}$ mass balance (MB) values, along the hair fibre.]

413 Individuals SI-T3 and SI-T32 present a comparable set of tendon collagen isotope values (respectively, $\delta^{13}\text{C}$
414 = -8.7‰ and -8.5‰ , $\delta^{15}\text{N}$ = $+15.5\text{‰}$ and $+16.1\text{‰}$, $\Delta^{13}\text{C}_{\text{Val-Phe}}$ = -1.7 and -0.9 , $\delta^{13}\text{C}$ Phe = -16.4‰ and $-$
415 17.1‰ , $\delta^{13}\text{C}$ Val = -18.1‰ and -18.0‰ , $\delta^{13}\text{C}$ Leu = -18.1‰ and -18.5‰ , $\delta^{13}\text{C}$ mass balance = -9.1‰ and $-$
416 9.0‰ , Table 2 and Fig. 9) that, in accordance with FRUITS diet estimates, indicate the predominant
417 consumption of C_4 plant products (56-69%), complemented with meat from terrestrial (14-34%) and marine
418 (3-9%) animals (Fig. 9). The less negative $\delta^{13}\text{C}$ values of both essential and non-essential amino acids (Fig.
419 9), compared to those of marine (SI-T74) and C_3 (SE-T3) resource consumers, show that the adults SI-T3
420 and SI-T32 gleaned the majority of their proteins, carbohydrates and lipids from crops and terrestrial animal
421 meat of C_4 origin. The keratin amino acid $\delta^{13}\text{C}$ values measured in the hair of SI-T3 and SI-T32 are also very
422 similar (respectively, $\delta^{13}\text{C}$ Phe = -15.1‰ to -17.0‰ and -14.6‰ to -16.6‰ , $\delta^{13}\text{C}$ Leu = -16.3‰ to -18.5‰
423 and -16.0‰ to -17.7‰ , $\delta^{13}\text{C}$ Val = -13.6‰ to -16.3‰ and -13.8‰ to -15.9‰ , $\delta^{13}\text{C}$ mass balance = -8.6‰
424 to -11.4‰ and -8.6‰ to -10.1‰ , Appendix C and Fig. 9). The limited range of both essential ($\leq 3\text{‰}$) and
425 non-essential ($< 5\text{‰}$) amino acid $\delta^{13}\text{C}$ values confirms that all three macronutrients (proteins, carbohydrates,
426 lipids) were predominantly retrieved from the same food source, such as C_4 resources. Given that the male
427 SI-T3 and female SI-T32 had a consistent dietary intake typical for mid-altitude (1,300 masl) inland
428 populations (based on local ecology), it may be speculated that these individuals were resident in the locality
429 of Pica, or moved across similar eco-zones, throughout approximately the last year of their lives. However,
430 FRUITS diet estimates (Tables 3-4 and Fig. 9), based on rib collagen isotope values ($\delta^{13}\text{C}$ = -10.4‰ , $\delta^{15}\text{N}$ =
431 $+21.0\text{‰}$) reported by Santana-Sagredo et al. (2015a), show that, during the previous 3 to 5 years, SI-T3
432 relied significantly on marine resources (22-38%), which contributed to 41-65% of his protein intake, and C_4
433 crops (40-48%). This dissimilarity between bone and tendon isotope values suggests that SI-T3 might have
434 recently entered the inland community after having lived in a location with full access to marine resources
435 (e.g. the Pacific coast and coastal valleys) or, less likely, that exotic foods were available at Pica (or
436 preferred by this individual) only during this period of time.

437 The tendon collagen isotope values of the adults SD-T24 and SF-T4 (respectively, $\delta^{13}\text{C}$ = -8.2‰ and -7.8‰ ,
438 $\delta^{15}\text{N}$ = $+16.2\text{‰}$ and $+17.5\text{‰}$, $\Delta^{13}\text{C}_{\text{Val-Phe}}$ = -0.8 and 0.0 , $\delta^{13}\text{C}$ Phe = -17.2‰ and -16.8‰ , $\delta^{13}\text{C}$ Val = -18.0‰
439 and -16.8‰ , $\delta^{13}\text{C}$ Leu = -18.5‰ and -18.5‰ , $\delta^{13}\text{C}$ mass balance = -8.9‰ and -8.5‰ , Table 2 and Fig. 9)
440 are broadly similar to those of the two aforementioned C_4 terrestrial resource consumers (SI-T3, SI-T32).
441 However, the keratin amino acid $\delta^{13}\text{C}$ values (Appendix C and Fig. 9) hint at a more diverse and complex
442 dietary intake. The range of amino acid $\delta^{13}\text{C}$ values measured in the hair of SF-T4 and SD-T24 is extensive,
443 being as high as $\sim 4\text{‰}$ - 5‰ for phenylalanine and leucine, and $\sim 8\text{‰}$ - 9‰ for alanine, proline, aspartic acid,
444 glutamic acid and arginine (for SD-T24). This, combined with the fact that along the hair fibres of SD-T24
445 and SF-T4 the $\delta^{13}\text{C}$ values of essential and non-essential amino acids change in a broadly synchronous
446 pattern (Fig. 9), suggests that all the macronutrients (proteins, lipids, carbohydrates) were sourced from a
447 variety of different foods that changed in proportion and quality over time. For instance, it may be inferred

448 that several months prior to death (4-5 cm hair segment), the young male SD-T24 had a significant intake of
449 C₃ foods, temporarily ceasing or lowering his consumption of C₄ resources, since the serine $\delta^{13}\text{C}$ value drops
450 to -8.7‰ , alanine to -15.5‰ , proline to -15.9‰ , arginine to -15.6‰ , aspartic acid to -13.5‰ , and glutamic
451 acid to -11.6‰ (Fig. 9). FRUITS dietary estimates (Fig. 9), calculated based upon rib collagen isotope
452 compositions published by Santana-Sagredo et al. (2015a), indicate that SD-T24 had a diet made up of
453 predominantly C₄ crops (50-65%) complemented by terrestrial animal meat (18-38%) and C₃ plant products
454 (10-13%) for at least several years before his death.

455 Although the adults SF-T4 and SD-T24 maintained a high intake of C₄ resources during approximately the
456 last 10 months and 3 to 5 years of life, respectively, they consumed additional and diverse foods (e.g. C₃ and,
457 to a lesser extent, marine resources) concentrated especially, but not only, during certain months. A potential
458 explanation is that these individuals, due to their social, economic or political status had access to a vast
459 array of resources or had control over the commercial traffic in the community. An alternative explanation is
460 that SF-T4 (female) and SD-T24 (male) had a high degree of mobility across different eco-zones from which
461 they derived their foods.

462 According to tendon collagen isotope data ($\delta^{13}\text{C} = -18.3\text{‰}$, $\delta^{15}\text{N} = +12.2\text{‰}$, $\Delta^{13}\text{C}_{\text{Val-Phe}} = -1.7$, $\delta^{13}\text{C Phe} = -$
463 25.9‰ , $\delta^{13}\text{C Val} = -27.6\text{‰}$, $\delta^{13}\text{C Leu} = -28.3\text{‰}$, Table 2 and Fig. 9), the young woman SE-T3 consumed
464 predominantly C₃ terrestrial resources during the final months of her life. The very negative $\delta^{13}\text{C}$ values of
465 both essential and non-essential amino acids (Fig. 9) indicate that she derived proteins, as well as
466 carbohydrates and lipids, from C₃ foods. FRUITS dietary estimates (Fig. 9) suggest that this young adult was
467 retrieving these three macronutrients mostly from plant products (77-79%) and also from some animal meat
468 (~11%). Based on the comparison of tendon and rib collagen isotope data (Table 2), her dietary habits were
469 broadly consistent at least during the last 3 to 5 years of life, although the increase in the $\delta^{15}\text{N}$ value, from
470 $+10.9\text{‰}$ in bone to $+12.2\text{‰}$ in tendon collagen, may suggest a greater consumption of meat from terrestrial
471 animals or, more likely, that she suffered an injury several months before death (D'Ortenzio et al., 2015,
472 Fuller et al., 2005, Reitsema, 2013), since instances of bone fractures have been identified.

473 The extensive ranges of keratin $\delta^{13}\text{C}$ values (Appendix C and Fig. 9), measured in both essential (e.g. 6.5‰
474 for valine and 5.8‰ for phenylalanine and leucine) and non-essential (e.g. 8‰ for serine, 7.2‰ for proline,
475 7‰ for arginine) amino acids, suggest that the SE-T3 female retrieved dietary proteins, carbohydrates and
476 lipids from different food sources: C₃ and C₄. However, the variations among the essential and non-essential
477 amino acid $\delta^{13}\text{C}$ values are broadly synchronous implying that one food source was swapped for the other
478 through time and that, if meat was consumed by SE-T3, these animals were fed on the same plant type (C₃ or
479 C₄) as that of the plant products directly consumed by this woman. Following the changes in amino acid $\delta^{13}\text{C}$
480 values along her hair fibre (Fig. 9), it can be inferred that SE-T3's diet was made up of mostly C₃ resources
481 about 10 months before death ($\delta^{13}\text{C Phe} = -21.2\text{‰}$, $\delta^{13}\text{C Leu} = -23.1\text{‰}$, $\delta^{13}\text{C Val} = -22.2\text{‰}$), but the
482 proportion of these foods declined gradually through time, being replaced by C₄ resources. However, during
483 a specific month (4-5 cm hair segment) the source of the carbohydrates was predominantly of C₄ origin (i.e.

484 less negative alanine, proline and glutamic acid $\delta^{13}\text{C}$ values). Closer to the time of death, her diet was mostly
485 composed of C_4 foods ($\delta^{13}\text{C}$ Phe= -15.8% , $\delta^{13}\text{C}$ Leu= -17.5% , $\delta^{13}\text{C}$ Val= -15.7%). The onset of a new diet
486 (i.e. rapid and drastic change) would have likely generated a sharp change in $\delta^{13}\text{C}$ values, followed by a
487 subsequent gradual adjustment to the new diet, based on previous studies (Ayliffe et al., 2004, O'Connell and
488 Hedges, 1999). However, this is not observed here, since the signal in SE-T3's hair is most likely the result
489 of a gradual change in diet. Nevertheless, the fact that only 10 cm of hair has been analysed makes it possible
490 that the change in diet happened before the time period investigated, and that the gradual change visible here
491 (Fig. 9) is, in reality, the isotope equilibration from a C_3 -based diet to the new C_4 -based diet.

492 It remains to be ascertained whether the female SE-T3 was experiencing a gradual transition towards C_4
493 resources as a result of moving from a place where C_3 foods were usually produced/collected and consumed,
494 such as the Precordillera and Altiplano, to the locality of Pica, where maize was more commonly grown and
495 eaten (Santana-Sagredo et al., 2015a, Uribe, 2006). The fact that tendon and rib collagens present C_3 isotope
496 signals might suggest that this young woman lived in another community, plausibly at a higher altitude,
497 which she left several months before death. The highland origin for this individual has already been proposed
498 by Santana-Sagredo et al. (2015a) based on the enamel $\delta^{18}\text{O}$ value (-11.3%) measured in the 3rd molar of
499 SE-T3. Furthermore, it cannot be excluded that this mobility pattern, spanning from the altiplano to
500 intermediate elevations, might be linked to camelid herding practices or long-distance trade, as women were
501 involved in these activities (Pomeroy, 2013). An alternative explanation, though less likely, is that SE-T3
502 was a local member of the Pica communities that had access to imported C_3 foods from the highlands, until
503 approximately a year before death but, during the final year of life, she was only able to access C_4 resources
504 such as maize, as a result of a change in her socio-economic status.

505 Given that the hair keratin recorded a transition from a C_3 to a C_4 food source, while the tendon tissues
506 reflect a completely C_3 dietary intake likely occurring during a previous period of time, it appears that the
507 tendon collagen is slower in turning over and adjusting to the new isotope signal than previously
508 hypothesised (Babraj et al., 2005). A recent study by Heinemeier et al. (2013) found that the renewal of
509 tendon tissues is quite variable within individuals, and that the turnover of the tendon core is very
510 limited/slow. With respect to the present study, it may be speculated that the tendon of SE-T3 had poor
511 regenerative capacity, or that only the core of the tendon has survived.

512 Based on tendon collagen isotope values ($\delta^{15}\text{N}$ = $+25.3\%$, $\delta^{13}\text{C}$ = -10.8% , $\Delta^{13}\text{C}_{\text{Val-Phe}}$ = 2.0 , $\delta^{13}\text{C}$ Phe= $-$
513 20.6% , $\delta^{13}\text{C}$ Val= -18.6% , $\delta^{13}\text{C}$ Leu= -19.9% , Table 2 and Fig. 9), it appears that towards the end of her
514 life the young woman SI-T74 relied predominantly on marine foodstuffs, which represented 41-51% of the
515 food she consumed and contributed to 71-81% of her protein intake (Tables 3-4). The valine $\delta^{13}\text{C}$ value of
516 this individual is in line with those of high-marine protein consumers reported by Honch et al. (2012), but the
517 $\delta^{13}\text{C}$ value of phenylalanine is slightly less negative, thus suggesting an additional intake of C_4 resources.
518 Dietary estimates generated by FRUITS models confirm the concurrent consumption of some C_4 crops (36-
519 42%), such as maize. According to tendon and rib collagen isotope compositions (Table 2), the dependence

520 on marine resources was likely consistent over the course of the last 3 to 5 years of SI-T74's life. The
521 slightly higher (+2.4‰) tendon $\delta^{15}\text{N}$ value (compared to that of bone) may have been induced by the
522 consumption of manured maize and/or high trophic level marine animals, such as sea lions, or alternatively
523 by the occurrence of a disease. Bone lesions have been identified on SI-T74's rib, which could have been
524 generated by tuberculosis infection, although there are many other potential causes (Roberts et al., 1994;
525 Santos and Roberts 2006).

526 The range of $\delta^{13}\text{C}$ values, measured along SI-T74's hair fibre, is greater than 4‰ for some essential amino
527 acids (phenylalanine, isoleucine, lysine) and greater than 6‰ for some non-essential amino acids (alanine,
528 glutamic acid, proline) (Appendix C and Fig. 9). This suggests that this young woman was deriving her
529 dietary proteins from a sole source of food, such as marine resources (based on the range of essential amino
530 acids), and that carbohydrates and lipids were also derived from another source, such as C_4 (based on the
531 range of non-essential amino acids). The variation in amino acid $\delta^{13}\text{C}$ values along the hair fibre (Fig. 9)
532 shows that the proportion of marine and C_4 foods changed in the diet of SI-T74 through time. Assuming a
533 growth rate of approximately 1 cm per month (Saitoh et al., 1969) for scalp hair in the anagen phase,
534 between about 10 to 8 months before her death this woman consumed predominantly marine resources
535 ($\Delta^{13}\text{C}_{\text{Val-Phe}} = 3.4$ to 4.3 , $\delta^{13}\text{C Phe} = -21.3\text{‰}$ to -19.6‰ , $\delta^{13}\text{C Val} = -17.3\text{‰}$ to -16.2‰ , $\delta^{13}\text{C Leu} = -20.1\text{‰}$ to
536 -19.1‰), while during the subsequent month (6-7 cm hair segment) she notably increased her intake of C_4
537 crops ($\Delta^{13}\text{C}_{\text{Val-Phe}} = 1.7$). This generated a shift of several per mill towards less negative $\delta^{13}\text{C}$ values in both
538 essential ($\delta^{13}\text{C Phe} =$ from -21.0‰ to -17.4‰ , $\delta^{13}\text{C Val} =$ from -17.3‰ to -15.7‰ , $\delta^{13}\text{C Leu} =$ from -19.5‰
539 to -17.6‰ , $\delta^{13}\text{C Lys} =$ from -13.7‰ to -11.8‰ , $\delta^{13}\text{C Ile} =$ from -13.2‰ to -11.2‰) and non-essential amino
540 acids ($\delta^{13}\text{C Ala} =$ from -14.1‰ to -8.0‰ , $\delta^{13}\text{C Pro} =$ from -14.6‰ to -8.4‰ , $\delta^{13}\text{C Glx} =$ from -8.7‰ to $-$
541 3.8‰). Subsequently, SI-T74 decreased her intake of C_4 resources, relying mostly on marine resources
542 ($\Delta^{13}\text{C}_{\text{Val-Phe}} = 3.0$) for a couple of months (4 to 6 cm hair segments). In the final period of her life, she then
543 returned to a diet rich in C_4 resources ($\Delta^{13}\text{C}_{\text{Val-Phe}} = 1.0$ to 1.8) and the $\delta^{13}\text{C}$ values of the aforementioned
544 amino acids again became less negative (Fig. 9). Considering that the human body breaks down and recycles
545 old proteins, as well as synthesizes new ones (O'Connell and Hedges, 1999), and that maize (the most likely
546 consumed C_4 resource) is low in protein (although this may be slightly increased by the use of fertilisers;
547 Keeney, 1970), it is reasonable to assume that her marine resource intake could be in reality lower than
548 previously discussed. In other words, the marine isotope signal recorded by the keratin amino acids may
549 derive from recycled proteins formed during a previous dietary phase characterized by high marine protein
550 consumption, and not from a recent intake of marine resources.

551 Given that Pica is situated ~80 km from the sea, it is difficult to explain this consistent and significant
552 consumption of marine resources throughout the years. Although some dried fish, fish bones and molluscs
553 have been found at the cemetery (Núñez, 1984), it is unclear how available they would have been for regular
554 consumption at such a distance from their source. The exchange network would need to have been developed
555 enough such that marine resources were available routinely and in high quantities for a few selected
556 individuals at Pica; it is therefore possible that SI-T74 had access to these foods, perhaps due to political,

557 cultural or socio-economic reasons. An alternative explanation is that she was resident on the coast or in the
558 coastal valleys of the Atacama Desert where marine foods and maize were both local, and only recently
559 before death she migrated to the locality of Pica where she was eventually buried, or was travelling inland
560 passing nearby these oases.

561

562 5. Conclusions

563 The individuals buried at Pica 8 present heterogeneous nutritional histories, both individually and
564 collectively. At the time of their deaths, all six adults (SD-T24, SI-T32, SF-T4, SI-T3, SE-T3, SI-T74) were
565 characterised by a terrestrial-C₄ diet, which is in line with what may have been the most easily accessible
566 foodstuffs in the locality of Pica: maize and terrestrial animal meat. Among these individuals, SI-T3 and SE-
567 T3 experienced a shift in their dietary intake several months to a year before death, possibly as a result of
568 relocation to mid-altitude communities. The original dietary habits of SE-T3 and SI-T3 were respectively
569 characteristic of the highlands (C₃ plants) and the coast and coastal valleys (marine resources). Only one
570 individual (SI-T74) might have been resident on the coast or in the coastal valleys of the Atacama Desert at
571 least over the course of the last 3 to 5 years of her life, based on a consistent intake of marine resources.
572 However, shortly before her death SI-T74 shifted to a more terrestrial diet when she possibly migrated to, or
573 was travelling close to, the locality of Pica.

574 Based on the dietary and mobility reconstruction of this subset of individuals, during the Late Intermediate
575 Period (~1,050-500 BP), the oasis of Pica appears to have been an economically and commercially dynamic
576 environment, which attracted people from distant regions. The individuals buried at Pica 8 may have been
577 actively involved in the trade of exotic objects and foodstuffs, acting as traders along the caravan routes, or
578 have belonged to the elite group who managed exchanges (Briones et al., 2005, Núñez, 1984, Pacheco and
579 Retamal, 2017, Pomeroy, 2013). In particular, the individuals from Sector I (SI-T3, SI-T74) may have been
580 relocated from, or linked to, communities located on the Pacific coast in order to support mutual
581 redistribution of resources between different eco-zones (Santana-Sagredo et al., 2015a, Uribe, 2006).

582 To our knowledge, this is the first study that has analysed collagen amino acid $\delta^{13}\text{C}$ values in tendon samples
583 from archaeological human remains, and this research shows that tendon may be a favourable substitute for
584 bone in palaeodietary reconstructions, providing it is preserved in the archaeological context. Given that
585 collagen is more abundant in tendon than in bone and its rate of turnover is faster (Babraj et al., 2005, Kjaer
586 et al., 2005), the dietary information reconstructed based on tendon stable isotope compositions presents a
587 higher temporal resolution and may be more fine-grained than that of bone collagen, since the original
588 isotope signal is averaged over a shorter period of time, i.e. several months/a year instead of years/decades.

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Table 1. LC gradient program for Primesep A column (2.1x250 mm, 100 Å, 5 µm).

Time (min)	Mobile phases (%)			Flow rate (µl/min)
Conditioning run	A	B	C	
0	0	92	8	110
35	0	92	8	110
36	100	0	0	110
55	100	0	0	110
Analytical run	A	B	C	
0	100	0	0	60
45	100	0	0	60
65	60	40	0	60
75	40	25	35	60
150	0	0	100	60
180	0	0	100	60

Table 2. Bulk carbon and nitrogen isotope compositions of collagens from Pica 8 individuals.

Individual			Tendon collagen (this study)					Rib collagen (Santana-Sagredo et al., 2015a)		Δ Rib collagen-tendon collagen	
Burial*	Sex	Age	%N	%C	C/N	δ ¹⁵ N/‰	δ ¹³ C/‰	δ ¹⁵ N/‰	δ ¹³ C/‰	δ ¹⁵ N/‰	δ ¹³ C/‰
SI-T74	F	20-35 yrs	15.9	46.1	3.4	+25.3 ± 0.0	-10.8 ± 0.0	+22.9	-10.5	-2.4	0.3
			16.1	46.0	3.3						
SD-T24	M	20-35 yrs	16.1	45.7	3.3	+16.2 ± 0.1	-8.2 ± 0.0	+14.2	-9.6	-2.0	-1.4
			16.1	45.6	3.3						
SI-T32	F	35-50 yrs	15.8	45.9	3.4	+16.1 ± 0.0	-8.5 ± 0.0				
			15.7	46.1	3.4						
SE-T3	F	20-35 yrs	16.1	46.6	3.4	+12.2 ± 0.1	-18.3 ± 0.0	+10.9	-18.6	-1.3	-0.3
			15.9	46.0	3.4						
SF-T4	F	35-50 yrs	16.5	45.6	3.2	+17.5 ± 0.0	-7.8 ± 0.0				
			16.0	44.2	3.2						
SI-T3	M	35-50 yrs	16.0	46.6	3.4	+15.5 ± 0.0	-8.7 ± 0.1	+21.0	-10.4	5.5	-1.7
			15.5	45.1	3.4						

*S indicates the burial sector; T indicates the grave number.

Table 3. FRUITS model diet estimates based on the collagen isotope data from the Pica 8 individuals.

Pica	SD-T24 Tendon		SD-T24 Bone		SE-T3 Tendon		SE-T3 Bone		SF-T4 Tendon	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Food (%)										
C3 plants	7	5	13	7	77	7	81	6	6	4
C4 plants	72	8	65	9	8	6	9	6	70	9
Terrestrial animals	11	8	18	11	11	8	7	6	9	7
Marine animals	10	4	4	3	4	3	3	2	15	5
Food fractions (%)										
Protein	27	3	29	4	30	2	27	2	28	3
Energy	73	3	71	3	70	2	73	2	72	3
Dietary proxies (Food)(%)										
$\delta^{13}\text{C}_{\text{col}}$ (C3 plants)	7	5	12	8	71	9	77	7	5	4
$\delta^{13}\text{C}_{\text{col}}$ (C4 plants)	61	10	54	11	7	5	8	5	59	11
$\delta^{13}\text{C}_{\text{col}}$ (Terr. animals)	18	11	28	15	17	12	11	8	14	9
$\delta^{13}\text{C}_{\text{col}}$ (Mar. animals)	14	6	6	4	5	4	4	3	22	7
$\delta^{15}\text{N}_{\text{col}}$ (C3 plants)	7	5	11	8	60	12	70	10	5	4
$\delta^{15}\text{N}_{\text{col}}$ (C4 plants)	39	12	34	12	4	3	5	4	37	11
$\delta^{15}\text{N}_{\text{col}}$ (Terr. animals)	30	17	45	20	27	16	19	13	23	14
$\delta^{15}\text{N}_{\text{col}}$ (Mar. animals)	24	9	10	7	9	6	6	5	35	9

Pica	SI-T3 Tendon		SI-T3 Bone		SI-T32 Tendon		SI-T74 Tendon		SI-T74 Bone	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Food (%)										
C3 plants	9	6	16	8	8	6	12	7	15	8
C4 plants	69	9	40	9	69	9	36	7	36	8
Terrestrial animals	15	9	6	5	14	9	1	2	3	3
Marine animals	7	4	38	7	9	4	51	4	46	5
Food fractions (%)										
Protein	28	3	40	2	29	3	43	1	42	1
Energy	72	3	60	2	71	3	57	1	58	1
Dietary proxies (Food)(%)										
$\delta^{13}\text{C}_{\text{col}}$ (C3 plants)	9	6	13	6	7	6	9	5	12	6
$\delta^{13}\text{C}_{\text{col}}$ (C4 plants)	58	11	29	7	58	11	24	5	25	6
$\delta^{13}\text{C}_{\text{col}}$ (Terr. animals)	22	13	9	6	21	13	3	2	5	4
$\delta^{13}\text{C}_{\text{col}}$ (Mar. animals)	11	5	49	7	14	6	64	4	58	6
$\delta^{15}\text{N}_{\text{col}}$ (C3 plants)	8	6	9	5	7	6	6	3	8	4
$\delta^{15}\text{N}_{\text{col}}$ (C4 plants)	37	12	14	5	36	12	10	3	12	4
$\delta^{15}\text{N}_{\text{col}}$ (Terr. animals)	37	19	12	9	34	18	3	3	6	5
$\delta^{15}\text{N}_{\text{col}}$ (Mar. animals)	18	9	65	8	23	9	81	4	74	6

Energy combines the contribution of lipids and carbohydrates. Food (%) represents the calorie contribution of each food group; Fraction (%) represents the calorie contribution of each food fraction; Dietary proxy (Food)(%) represents the calorie contribution of each food group to the dietary proxies. Estimates are normalised to 100%, and uncertainty is 1-sigma (Fernandes et al., 2014, Fernandes, 2015, Fernandes et al., 2015).

Table 4. FRUITS model diet estimates based on the collagen isotope data from the Pica 8 individuals (Model with ‘maize fertilized with guano’).

Pica	SD-T24 Tendon		SD-T24 Bone		SE-T3 Tendon		SE-T3 Bone		SF-T4 Tendon	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Food (%)										
C3 plants	6	5	10	7	79	7	84	6	5	5
Maize manured (guano)	59	7	50	6	7	5	5	4	64	7
Terrestrial animals	31	8	38	7	11	8	9	7	27	7
Marine animals	4	3	2	2	3	3	2	2	4	4
Food fractions (%)										
Protein	36	3	39	2	30	2	29	2	33	3
Energy	64	3	61	2	70	2	71	2	67	3
Dietary proxies (Food)(%)										
$\delta^{13}\text{C}_{\text{col}}$ (C3 plants)	5	5	8	6	73	9	79	8	5	4
$\delta^{13}\text{C}_{\text{col}}$ (Maize manured)	44	7	36	6	6	4	5	4	49	8
$\delta^{13}\text{C}_{\text{col}}$ (Terr. animals)	46	9	53	8	16	11	13	10	40	8
$\delta^{13}\text{C}_{\text{col}}$ (Mar. animals)	5	4	3	3	5	4	3	3	6	5
$\delta^{15}\text{N}_{\text{col}}$ (C3 plants)	4	4	6	5	63	12	70	12	4	4
$\delta^{15}\text{N}_{\text{col}}$ (Maize manured)	21	5	15	4	4	3	3	2	25	6
$\delta^{15}\text{N}_{\text{col}}$ (Terr. animals)	68	9	75	7	25	15	22	14	62	9
$\delta^{15}\text{N}_{\text{col}}$ (Mar. animals)	7	6	4	4	8	6	5	4	9	7

Pica	SI-T3 Tendon		SI-T3 Bone		SI-T32 Tendon		SI-T74 Tendon		SI-T74 Bone	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Food (%)										
C3 plants	7	5	18	9	6	5	14	7	18	7
Maize manured (guano)	56	7	48	11	57	7	42	10	45	12
Terrestrial animals	34	7	12	7	33	7	3	2	6	5
Marine animals	3	3	22	10	4	3	41	9	31	11
Food fractions (%)										
Protein	38	3	35	3	37	3	39	2	36	3
Energy	62	2	65	3	63	2	61	2	64	3
Dietary proxies (Food)(%)										
$\delta^{13}\text{C}_{\text{col}}$ (C3 plants)	6	5	16	8	5	4	11	5	15	6
$\delta^{13}\text{C}_{\text{col}}$ (Maize manured)	41	7	38	12	42	7	31	10	35	12
$\delta^{13}\text{C}_{\text{col}}$ (Terr. animals)	49	8	16	10	48	8	4	3	8	6
$\delta^{13}\text{C}_{\text{col}}$ (Mar. animals)	4	4	30	13	5	4	54	10	42	13
$\delta^{15}\text{N}_{\text{col}}$ (C3 plants)	4	4	13	8	4	4	8	4	12	6
$\delta^{15}\text{N}_{\text{col}}$ (Maize manured)	19	5	21	10	20	5	16	8	19	10
$\delta^{15}\text{N}_{\text{col}}$ (Terr. animals)	71	8	25	13	70	8	5	5	12	9
$\delta^{15}\text{N}_{\text{col}}$ (Mar. animals)	6	5	41	16	6	5	71	10	57	15

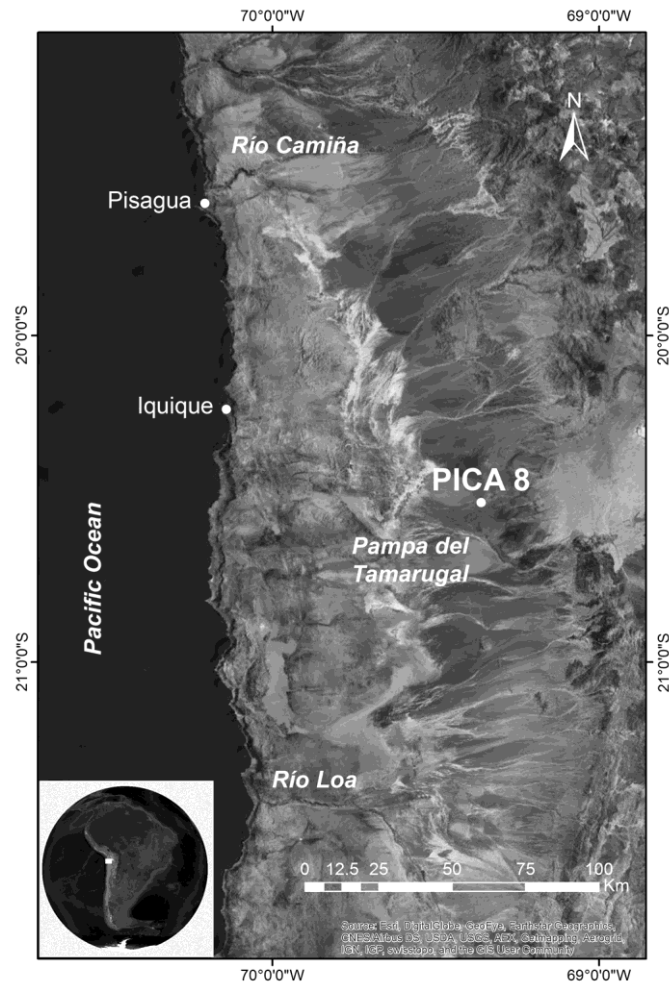


Fig 1. Map of northern Chile showing the location of the Pica 8 site.

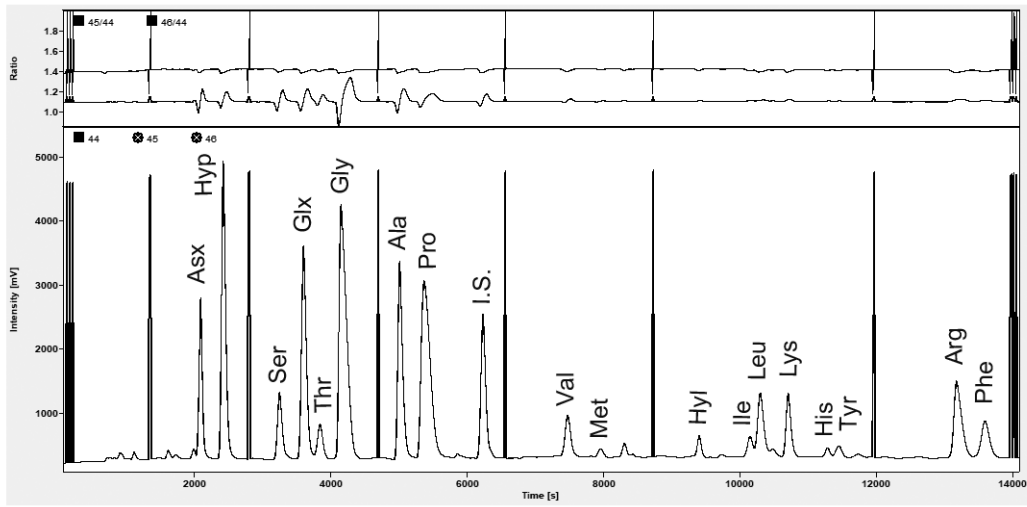


Fig 2. LC-IRMS chromatogram of tendon collagen hydrolysate (from individual SE-T3).

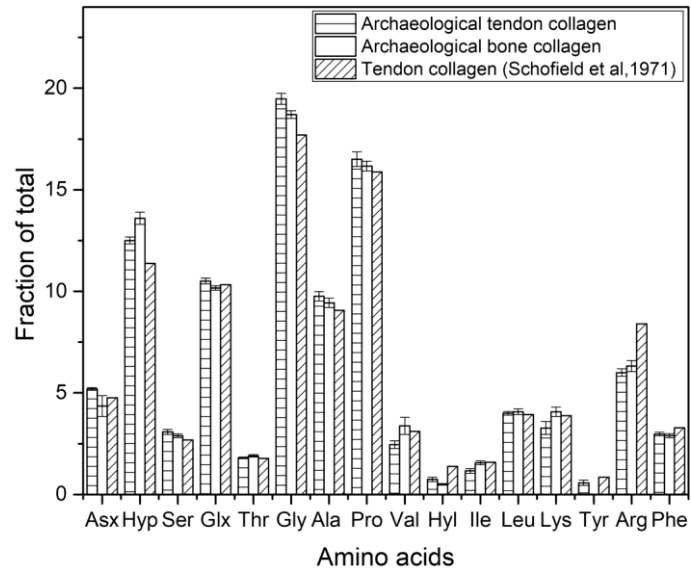


Fig 3. Fractions of the total (%) of amino acid peak areas measured in archaeological tendon collagen (this study) and bone collagen (mean \pm 1 σ), and of amino acid carbon weights (%) of human tendon collagen derived from Schofield et al. (1971).

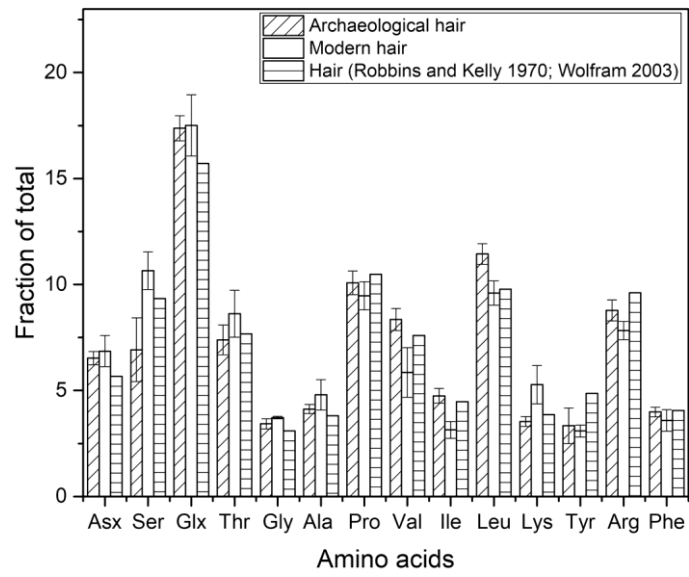


Fig 4. Fractions of the total (%) of amino acid peak areas measured in archaeological and modern hair (mean \pm 1 σ), and of amino acid carbon weights (%) measured in human hair (Robbins and Kelly, 1970; Wolfram, 2003).

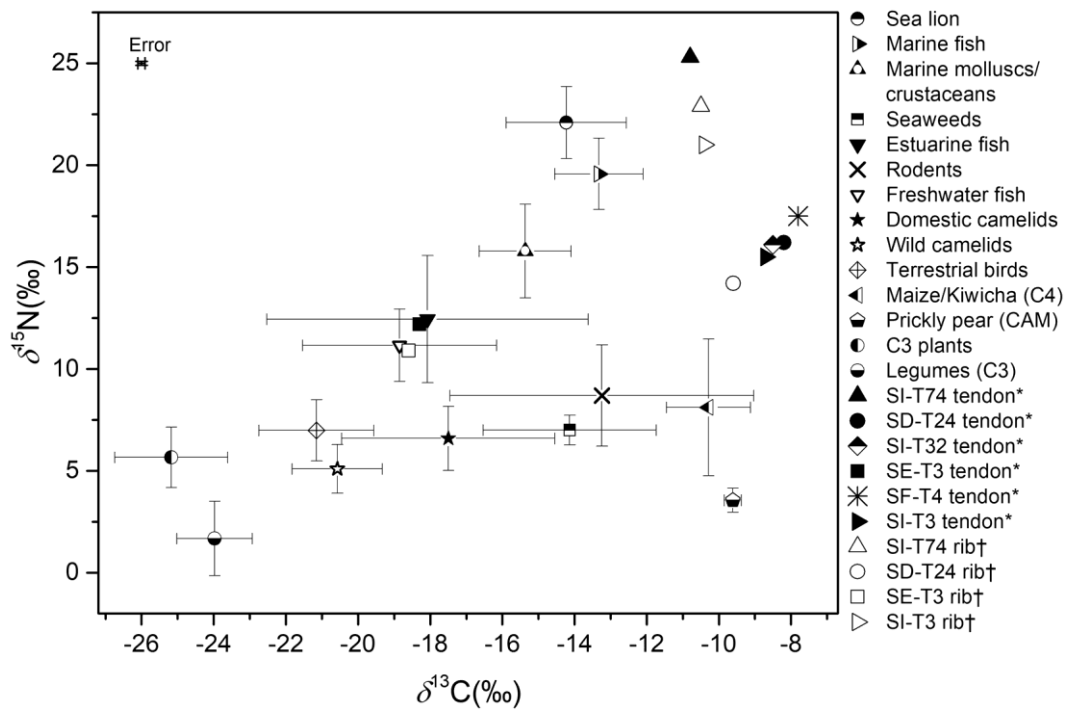


Fig 5. Plot of $\delta^{15}\text{N}$ values versus $\delta^{13}\text{C}$ values of tendon collagen (*, this study) and bone collagen (†, Santana-Sagredo et al., 2015a) from Pica 8 individuals and of the edible portion of South American flora and fauna.

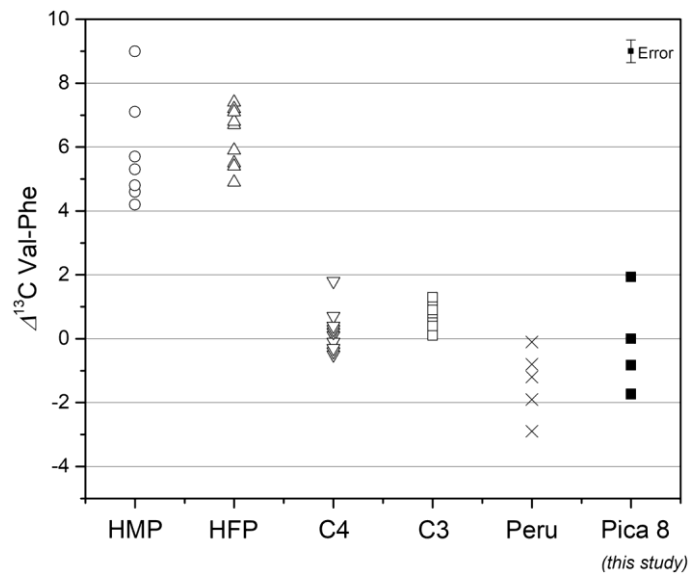


Fig 6. Plot of $\Delta^{13}\text{C}_{\text{Val-Phe}}$ values for tendon collagen (this study) and bone collagen (Honch et al., 2012). HMP = high-marine protein consumer; HFP = high-freshwater protein consumer; C4=C4 plant consumers; C3=C3 plant consumers; Peru = mixed-diet group, Peru, Huari AD 500–900.

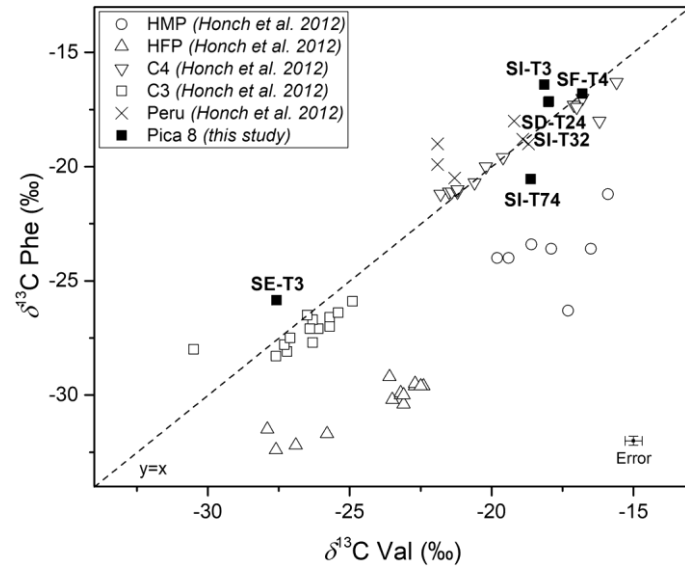


Fig 7. Plot of $\delta^{13}\text{C}$ phenylalanine values vs. $\delta^{13}\text{C}$ valine values for tendon collagen (this study) and bone collagen (Honch et al., 2012).

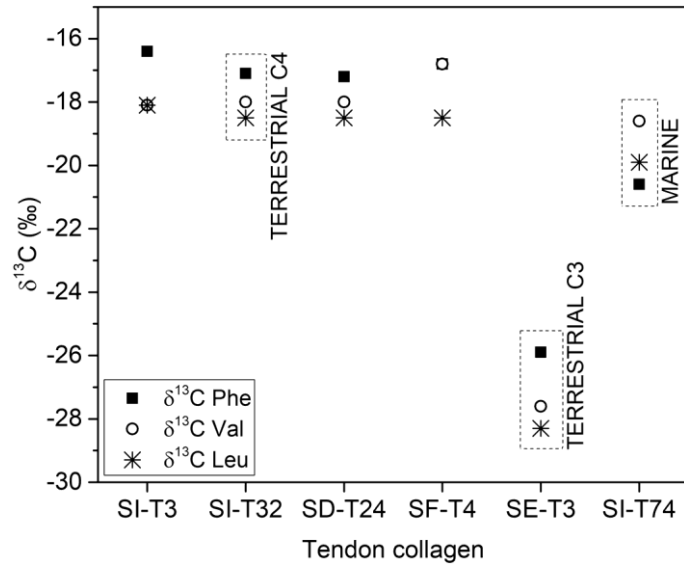
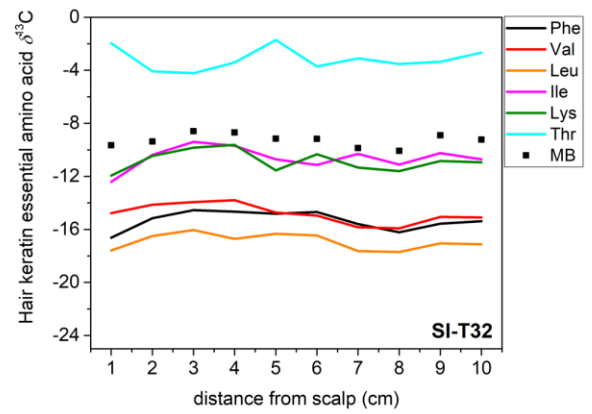
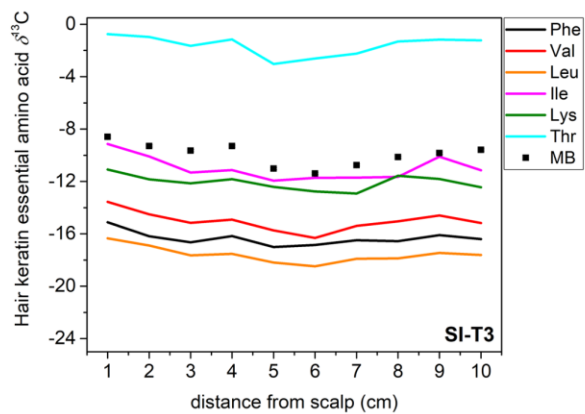
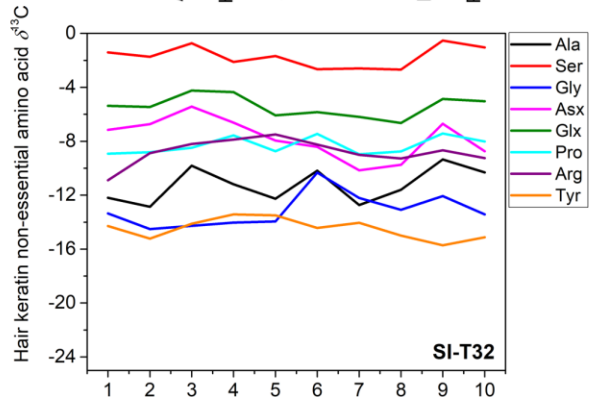
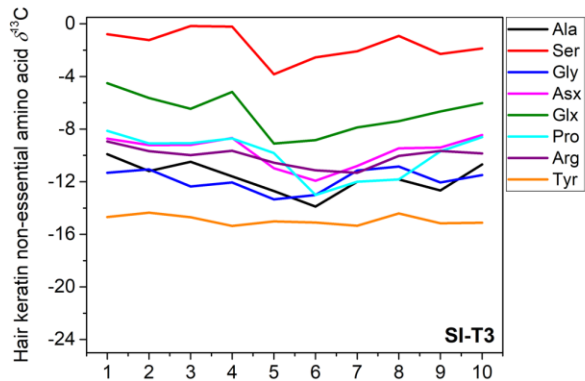
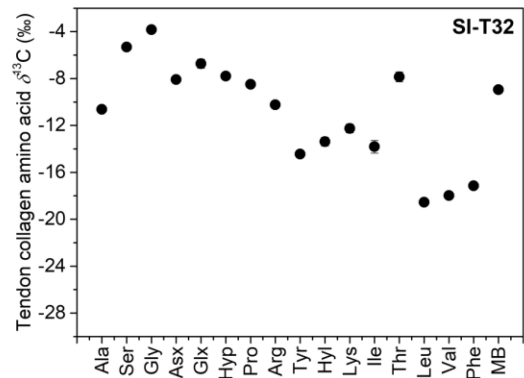
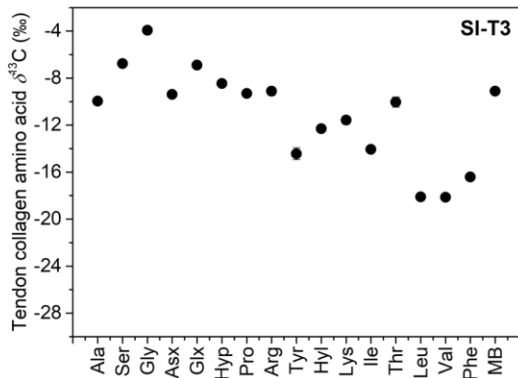
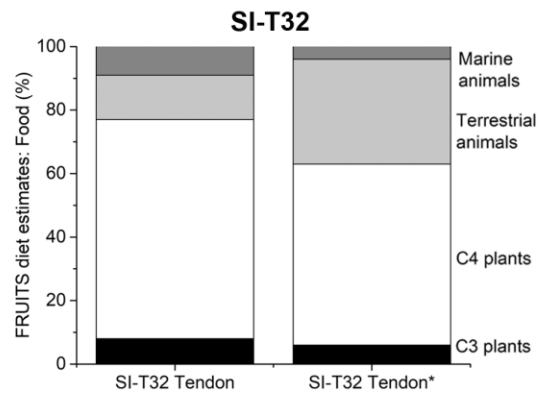
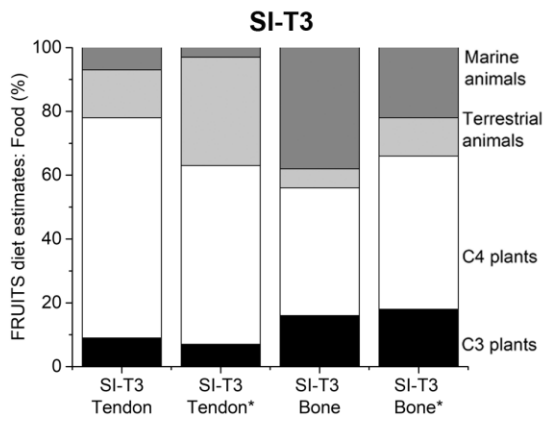
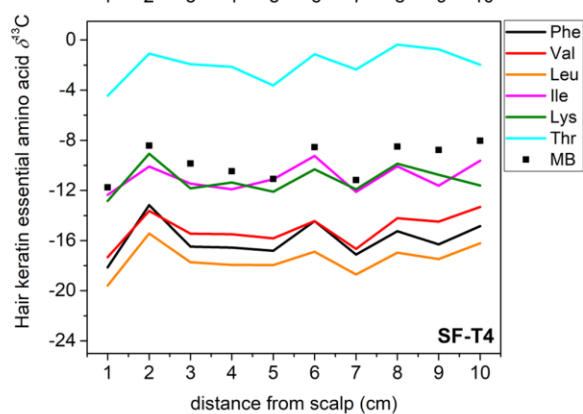
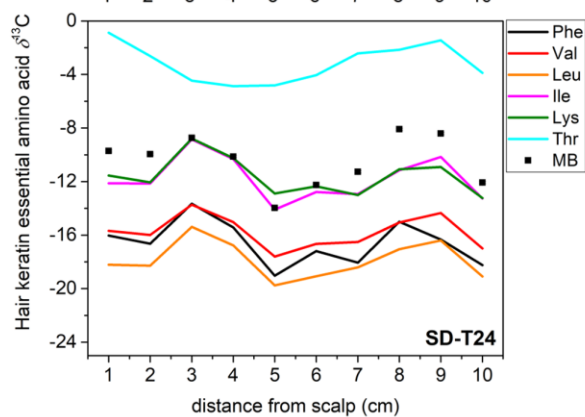
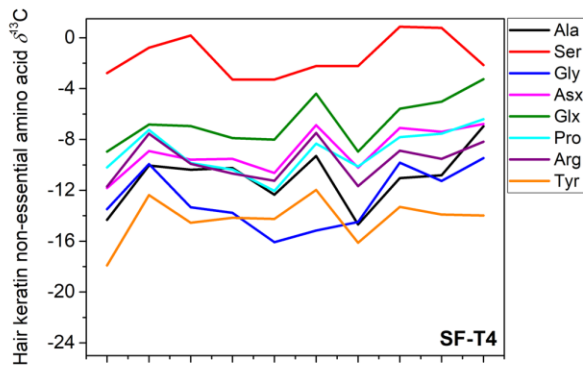
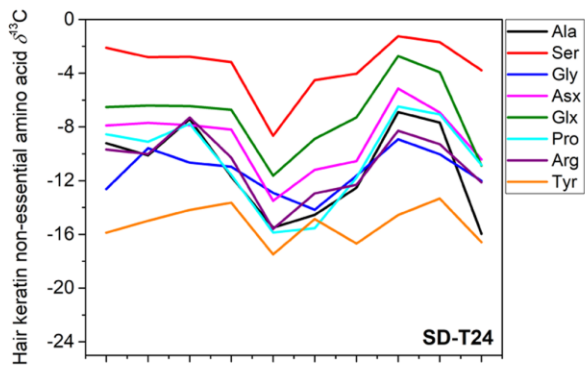
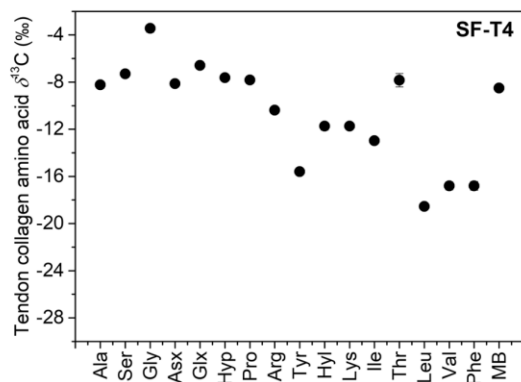
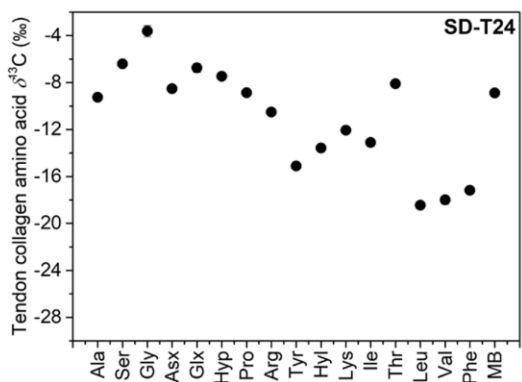
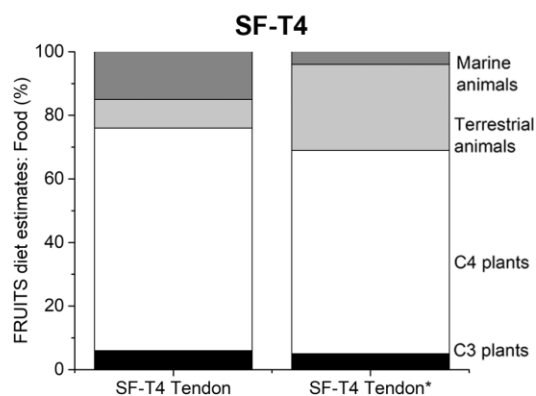
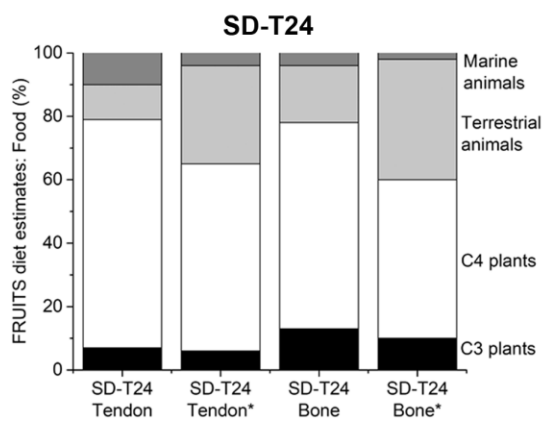


Fig 8. Proposed model for assessing the origin of the predominant dietary intake (terrestrial vs. marine), based on tendon collagen amino acid $\delta^{13}\text{C}$ values.





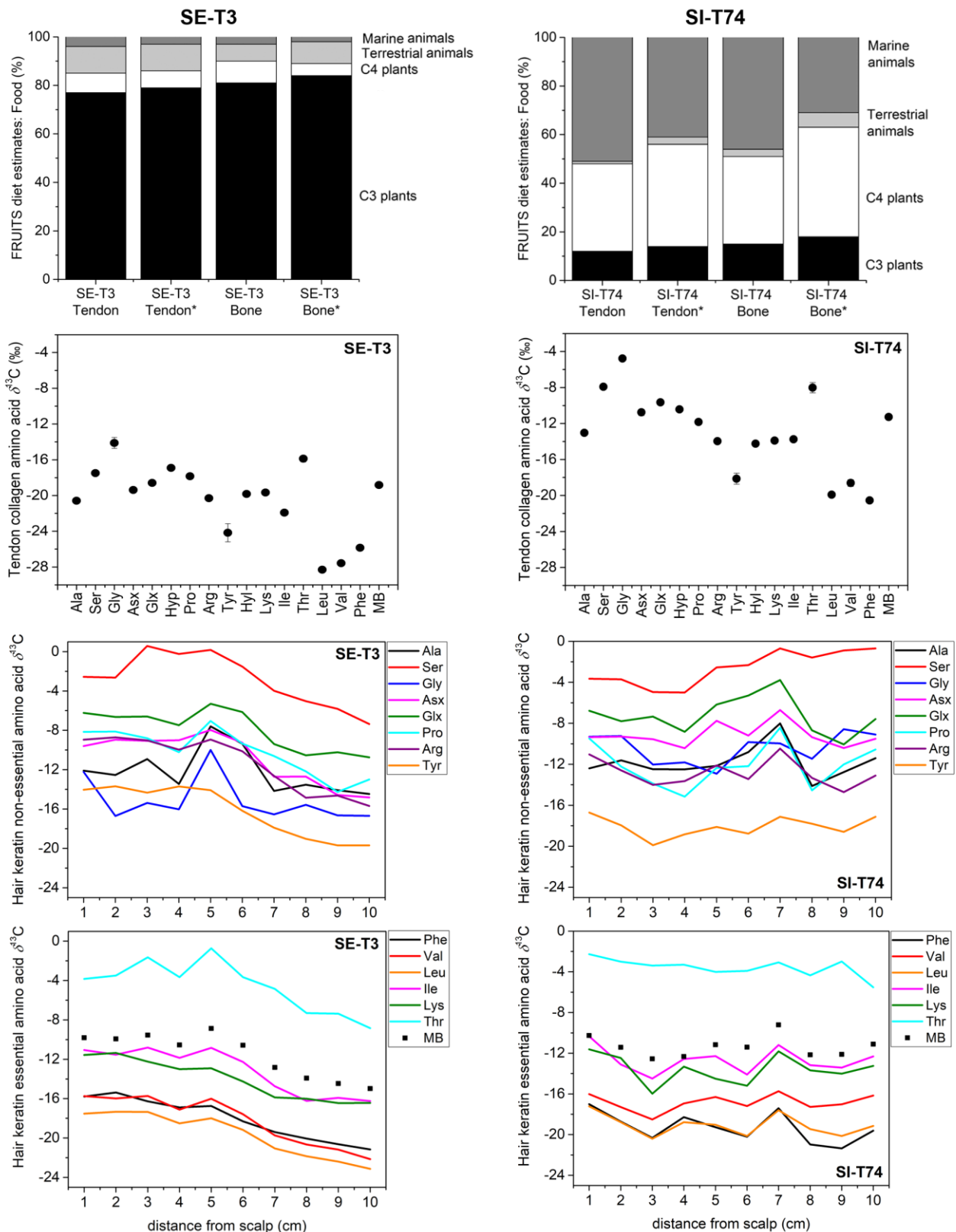


Fig 9. For each individual, from top to bottom: Calorie contribution of each food group to the diet, estimated via Bayesian stable isotope mixing model FRUITS, based on bulk $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ values of tendon collagen (this study) and bone collagen (Santana-Sagredo et al., 2015a). (Asterisk indicates FRUITS model with ‘maize fertilised with guano’ replacing ‘C4 plants’ food group); Amino acid $\delta^{13}\text{C}$ values and calculated $\delta^{13}\text{C}$ mass balance (MB) values for tendon collagen; Keratin $\delta^{13}\text{C}$ values of non-essential and essential amino acids, and calculated $\delta^{13}\text{C}$ mass balance (MB) values, along the hair fibre.

Appendix A. Supplementary data

Bayesian stable isotope mixing model: FRUITS

The Bayesian mixing model Food Reconstruction Using Isotopic Transferred Signals (FRUITS) (Fernandes et al., 2014) has been applied to the tendon collagen isotope data produced in this study and to the bone collagen isotope data published by Santana-Sagredo et al. (2015) in order to achieve an estimation of the qualitative and quantitative nutritional intake of the Pica individuals.

The dietary proxies used in the FRUITS models are the tendon and rib collagen $\delta^{15}\text{N}$ and $\delta^{13}\text{C}$ values with an associated uncertainty of 0.5‰ to account for instrumental analytical error. The food groups used include the widest array of foods available through trade from the Pacific coast, coastal valleys, mid-altitude valleys and highlands. The food groups consist of: (1) C4 plants (cultivated and wild), (2) marine animals (fish, sea lions, shellfish), (3) C3 plants (fruits, vegetables, legumes), and (4) terrestrial animals (camelids, rodents and birds) consuming either C3, C4 or mixed C3-C4 plants (i.e. wild and domesticated animals living at different elevations) (Mora et al., 2017). To account for the use of guano (seabird dung) for maize cultivation, a second model was run, substituting the ‘C4 plants’ food group with ‘maize manured with guano’ (Table A.1).

Table A.1. Isotope values and concentrations of food fractions for each food group used in the FRUITS models.

Food group	Food fraction	$\delta^{13}\text{C}/\text{‰}$	$\delta^{15}\text{N}/\text{‰}$	Concentration (%)
C3 plants	Protein	-26.7 ± 1	$+4.1\pm 1$	23 ± 2.5
	Energy [†]	-24.2 ± 1		77 ± 2.5
C4 plants*	Protein	-12.3 ± 1	$+8.1\pm 1$	14 ± 2.5
	Energy [†]	-9.8 ± 1		86 ± 2.5
Terrestrial animals	Protein	-17.7 ± 1	$+8.9\pm 1$	78 ± 2.5
	Energy [†]	-23.7 ± 1		22 ± 2.5
Marine animals	Protein	-13.5 ± 1	$+19.2\pm 1$	68 ± 2.5
	Energy [†]	-19.5 ± 1		32 ± 2.5
*Substituting food group:				
Maize fertilised with guano	Protein	-11.8 ± 1	$+22.8\pm 1$	11 ± 2.5
	Energy [†]	-9.3 ± 1		89 ± 2.5

[†] Energy combines the contribution of lipids and carbohydrates

Details on calculations of isotope values and concentrations of protein, lipid and carbohydrate for each food group were reported in Mora et al. (2017) and were based on Fernandes (2015) and Newsome et al. (2004). As in Mora et al. (2017), the carbon contribution from dietary proteins has been set below 45% by applying an a priori constraint to the FRUITS model. The diet-to-tissue offset for human bone collagen has been estimated by Fernandes et al. (2012) via regression analysis performed on isotope data measured in controlled feeding experiments on animals. The statistical analysis showed that carbon in collagen is routed by about $74\pm 4\%$ from dietary proteins and by 26% from the energetic macronutrients (lipids and

carbohydrates), while nitrogen in collagen is derived from only dietary proteins (100%) (Fernandes et al., 2012, Fernandes, 2015). The resulting diet-to-collagen offsets, which have been proposed by Fernandes et al. (2012, 2015) and used by several authors (Andrade et al., 2015, Fernandes et al., 2015), are: $4.8 \pm 0.5\%$ for $\delta^{13}\text{C}$ and $5.5 \pm 0.5\%$ for $\delta^{15}\text{N}$ values. Given that the contribution of the energetic macronutrients to the (bone) collagen carbon is from the three glycolytic amino acids (serine, glycine, alanine) (Fernandes et al., 2012) and that the amino acid composition of bone and tendon collagen is similar (if not identical, being both composed of type I collagen) (Eastoe, 1955), the same diet-to-collagen offsets and macronutrients' contributions will be used in the present model for the collagens extracted from bones and tendons.

It is acknowledged that it is not possible to take into account the isotope fractionation that might exist associated with the different protein metabolism and remodelling between bone and tendon. Furthermore, these FRUITS models do not account for possible alterations of the healthy metabolism induced by malnutrition or disease.

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Appendix B. Supplementary data

Amino acid $\delta^{13}\text{C}$ (‰) values (in order of LC elution), calculated $\delta^{13}\text{C}$ (‰) Mass Balance (MB) values and bulk $\delta^{13}\text{C}$ (‰) values for tendon collagen.

	Asx	Hyp	Ser	Glx	Thr	Gly	Ala	Pro	Val	Hyl	Ile	Leu	Lys	Tyr	Arg	Phe	MB	Bulk
SI-T74	-11.0	-10.5	-7.8	-9.9	-7.6	-4.9	-13.0	-12.0	-18.9	-14.1	-13.6	-19.8	-13.7	-17.7	-13.8	-20.6		
SI-T74	-10.5	-10.3	-8.0	-9.4	-8.4	-4.7	-13.1	-11.7	-18.3	-14.4	-13.9	-20.0	-14.0	-18.6	-14.2	-20.5		
mean	-10.8	-10.4	-7.9	-9.7	-8.0	-4.8	-13.0	-11.8	-18.6	-14.2	-13.7	-19.9	-13.9	-18.1	-14.0	-20.6	-11.3	-10.8
SD	0.3	0.2	0.1	0.3	0.6	0.1	0.0	0.2	0.4	0.2	0.2	0.2	0.2	0.6	0.3	0.1		
SD-T24	-8.3	-7.2	-6.5	-6.5	-7.9	-3.3	-9.1	-8.8	-18.2	-13.6	-13.3	-18.4	-12.2	-15.0	-10.5	-17.2		
SD-T24	-8.7	-7.7	-6.3	-7.0	-8.3	-4.0	-9.4	-9.0	-17.7	-13.6	-12.9	-18.5	-11.9	-15.2	-10.5	-17.2		
mean	-8.5	-7.5	-6.4	-6.7	-8.1	-3.6	-9.2	-8.9	-18.0	-13.6	-13.1	-18.5	-12.1	-15.1	-10.5	-17.2	-8.9	-8.2
SD	0.3	0.3	0.2	0.4	0.2	0.5	0.2	0.2	0.4	0.0	0.3	0.1	0.2	0.2	0.0	0.0		
SI-T32	-8.2	-8.0	-5.2	-7.0	-8.2	-3.7	-10.6	-8.5	-18.1	-13.7	-13.4	-18.3	-12.0		-10.2	-17.0		
SI-T32	-7.9	-7.6	-5.5	-6.5	-7.6	-4.0	-10.6	-8.4	-17.9	-13.1	-14.2	-18.7	-12.5	-14.4	-10.3	-17.3		
mean	-8.1	-7.8	-5.3	-6.7	-7.9	-3.8	-10.6	-8.5	-18.0	-13.4	-13.8	-18.5	-12.3	-14.4	-10.2	-17.1	-9.0	-8.5
SD	0.2	0.3	0.2	0.4	0.4	0.3	0.0	0.1	0.1	0.4	0.5	0.3	0.4		0.0	0.2		
SE-T3	-19.6	-17.1	-17.7	-18.4	-15.6	-13.7	-20.5	-17.7	-27.8	-19.6	-21.8	-28.4	-19.7	-23.5	-20.3	-25.7		
SE-T3	-19.2	-16.8	-17.3	-18.8	-16.1	-14.5	-20.7	-18.0	-27.4	-20.0	-22.1	-28.2	-19.6	-24.9	-20.3	-26.0		
mean	-19.4	-16.9	-17.5	-18.6	-15.9	-14.1	-20.6	-17.9	-27.6	-19.8	-21.9	-28.3	-19.7	-24.2	-20.3	-25.9	-18.8	-18.3
SD	0.3	0.2	0.2	0.3	0.4	0.6	0.2	0.2	0.3	0.3	0.2	0.1	0.1	1.0	0.0	0.3		
SF-T4	-8.3	-7.7	-7.3	-6.6	-8.2	-3.7	-8.0	-8.0	-17.1	-12.0	-12.8	-18.7	-11.9	-15.6	-10.5	-17.1		
SF-T4	-7.9	-7.6	-7.4	-6.6	-7.4	-3.2	-8.4	-7.7	-16.6	-11.5	-13.1	-18.4	-11.6		-10.2	-16.5		
mean	-8.1	-7.6	-7.3	-6.6	-7.8	-3.4	-8.2	-7.8	-16.8	-11.7	-13.0	-18.5	-11.7	-15.6	-10.4	-16.8	-8.5	-7.8
SD	0.3	0.1	0.1	0.0	0.6	0.3	0.3	0.2	0.3	0.3	0.2	0.3	0.2		0.2	0.4		
SI-T3	-9.6	-8.6	-6.9	-7.0	-10.4	-3.7	-9.9	-9.4	-18.3	-12.2	-14.1	-18.2	-11.8	-14.8	-9.4	-16.6		
SI-T3	-9.2	-8.3	-6.6	-6.8	-9.7	-4.2	-10.0	-9.2	-18.0	-12.4	-14.0	-18.0	-11.3	-14.1	-8.9	-16.2		
mean	-9.4	-8.5	-6.8	-6.9	-10.0	-3.9	-10.0	-9.3	-18.1	-12.3	-14.1	-18.1	-11.6	-14.4	-9.1	-16.4	-9.1	-8.7
SD	0.3	0.2	0.2	0.1	0.4	0.3	0.1	0.1	0.2	0.2	0.1	0.2	0.3	0.5	0.3	0.3		

Appendix C. Supplementary data

Table C.1. Amino acid $\delta^{13}\text{C}$ values in order of LC elution and calculated $\delta^{13}\text{C}$ Mass Balance (MB) values for hair keratin (1cm segment sequentially cut along the hair fibre starting at the root).

Individual	cm	Asx	Ser	Glx	Thr	Gly	Ala	Pro	Val	Ile	Leu	Lys	Tyr	Arg	Phe	MB
SI-T74	1	-9.2	-3.9	-6.8	-2.5	-9.8	-12.5	-9.6	-15.9	-10.4	-17.5	-11.8	-16.4	-11.3	-17.2	-10.3
SI-T74	1	-9.5	-3.5	-6.8	-2.0	-8.8	-12.3	-9.3	-16.1	-10.3	-17.0	-11.4	-17.0	-10.8	-16.8	
SI-T74	mean	-9.4	-3.7	-6.8	-2.3	-9.3	-12.4	-9.5	-16.0	-10.3	-17.2	-11.6	-16.7	-11.0	-17.0	
	SD	0.2	0.3	0.0	0.4	0.7	0.2	0.2	0.2	0.0	0.3	0.3	0.4	0.4	0.3	
SI-T74	2	-9.5	-3.7	-8.0	-3.3	-8.9	-11.8	-12.0	-17.2	-12.9	-18.8	-12.7	-18.0	-12.6	-18.9	-11.4
SI-T74	2	-9.1	-3.7	-7.6	-2.7	-9.6	-11.5	-12.4	-17.4	-13.3	-18.7	-12.3	-18.0	-12.6	-18.6	
SI-T74	mean	-9.3	-3.7	-7.8	-3.0	-9.2	-11.6	-12.2	-17.3	-13.1	-18.8	-12.5	-18.0	-12.6	-18.7	
	SD	0.3	0.1	0.3	0.4	0.6	0.2	0.3	0.2	0.3	0.1	0.3	0.0	0.0	0.2	
SI-T74	3	-9.8	-4.9	-7.3	-3.6	-12.5	-12.7	-14.2	-18.8	-14.3	-20.6	-15.7	-19.6	-14.0	-20.3	-12.5
SI-T74	3	-9.3	-5.0	-7.4	-3.1	-11.6	-12.3	-13.6	-18.2	-14.7	-20.2	-16.3	-20.2	-14.0	-20.3	
SI-T74	mean	-9.6	-5.0	-7.3	-3.4	-12.0	-12.5	-13.9	-18.5	-14.5	-20.4	-16.0	-19.9	-14.0	-20.3	
	SD	0.3	0.1	0.1	0.3	0.6	0.3	0.4	0.4	0.3	0.3	0.4	0.4	0.0	0.0	
SI-T74	4	-10.4	-5.0	-8.7	-3.7	-11.5	-12.4	-15.3	-17.0	-12.4	-19.0	-13.5	-18.8	-13.9	-18.4	-12.3
SI-T74	4	-10.5	-5.0	-9.0	-2.9	-12.1	-12.6	-15.1	-16.9	-12.8	-18.6	-13.1		-13.4	-18.1	
SI-T74	mean	-10.4	-5.0	-8.8	-3.3	-11.8	-12.5	-15.2	-16.9	-12.6	-18.8	-13.3	-18.8	-13.6	-18.3	
	SD	0.1	0.0	0.2	0.6	0.4	0.1	0.1	0.1	0.3	0.3	0.3		0.3	0.2	
SI-T74	5	-7.7	-2.4	-6.3	-3.6	-12.8	-12.0	-12.0	-16.0	-11.9	-18.7	-14.2	-18.1	-11.9	-19.0	-11.2
SI-T74	5	-7.8	-2.7	-6.1	-4.5	-13.1	-12.3	-12.7	-16.6	-12.7	-19.3	-14.8		-12.3	-19.6	
SI-T74	mean	-7.8	-2.5	-6.2	-4.0	-12.9	-12.2	-12.3	-16.3	-12.3	-19.0	-14.5	-18.1	-12.1	-19.3	
	SD	0.1	0.2	0.1	0.6	0.2	0.2	0.5	0.5	0.5	0.4	0.4		0.3	0.4	
SI-T74	6	-9.1	-2.3	-5.6	-3.8	-10.0	-10.8	-12.0	-16.9	-14.3	-20.2	-15.0	-18.5	-13.5	-20.5	-11.4
SI-T74	6	-9.3	-2.4	-5.0	-4.0	-9.7	-10.8	-12.4	-17.4	-13.9	-20.0	-15.4	-19.1	-13.4	-19.9	
SI-T74	mean	-9.2	-2.3	-5.3	-3.9	-9.8	-10.8	-12.2	-17.2	-14.1	-20.1	-15.2	-18.8	-13.4	-20.2	
	SD	0.2	0.0	0.4	0.2	0.2	0.0	0.3	0.4	0.2	0.1	0.2	0.4	0.1	0.4	
SI-T74	7	-6.6	-0.5	-3.8	-2.6	-9.6	-7.8	-8.2	-15.5	-11.0	-17.4	-11.5	-17.1	-10.3	-17.2	-9.2
SI-T74	7	-6.8	-0.9	-3.7	-3.5	-10.3	-8.2	-8.7	-16.0	-11.4	-17.8	-12.1		-10.7	-17.6	
SI-T74	mean	-6.7	-0.7	-3.8	-3.1	-10.0	-8.0	-8.4	-15.7	-11.2	-17.6	-11.8	-17.1	-10.5	-17.4	
	SD	0.2	0.3	0.1	0.6	0.5	0.3	0.3	0.4	0.3	0.2	0.4		0.3	0.3	
SI-T74	8	-9.2	-1.8	-9.0	-4.8	-11.0	-14.5	-14.8								
SI-T74	8	-9.5	-1.4	-8.4	-3.9	-11.9	-13.7	-14.3	-17.3	-13.2	-19.5	-13.7	-17.8	-13.3	-21.0	

SI-T74	mean	-9.3	-1.6	-8.7	-4.3	-11.5	-14.1	-14.6	-17.3	-13.2	-19.5	-13.7	-17.8	-13.3	-21.0	-12.2
	SD	0.2	0.3	0.4	0.6	0.7	0.6	0.3								
SI-T74	9	-10.2	-1.0	-9.9	-2.7	-9.1	-12.7	-12.1	-17.0	-13.2	-20.1	-13.8	-18.8	-14.7	-21.4	
SI-T74	9	-10.6	-0.8	-10.2	-3.3	-8.1	-12.8	-11.9	-17.0	-13.6	-20.2	-14.2	-18.4	-14.8	-21.3	
SI-T74	mean	-10.4	-0.9	-10.1	-3.0	-8.6	-12.8	-12.0	-17.0	-13.4	-20.1	-14.0	-18.6	-14.7	-21.3	-12.1
	SD	0.2	0.2	0.2	0.5	0.7	0.1	0.2	0.0	0.3	0.0	0.3	0.2	0.1	0.1	
SI-T74	10	-9.6	-0.9	-7.6	-5.5	-9.5	-11.6	-10.9	-16.3	-12.6	-19.3	-13.5	-16.8	-13.3	-19.7	
SI-T74	10	-9.4	-0.5	-7.5	-5.5	-8.7	-11.2	-10.3	-16.0	-12.0	-19.0	-13.0	-17.4	-12.9	-19.6	
SI-T74	mean	-9.5	-0.7	-7.6	-5.5	-9.1	-11.4	-10.6	-16.2	-12.3	-19.1	-13.2	-17.1	-13.1	-19.6	-11.1
	SD	0.1	0.2	0.1	0.0	0.6	0.3	0.4	0.2	0.4	0.2	0.4	0.4	0.2	0.1	
SD-T24	1	-7.7	-2.2	-6.7	-0.8	-12.3	-9.1	-8.7	-15.7	-12.4	-18.5	-11.8	-16.0	-10.0	-16.1	
SD-T24	1	-8.1	-2.0	-6.3	-0.9	-13.0	-9.4	-8.4	-15.7	-11.9	-17.9	-11.3	-15.8	-9.4	-16.0	
SD-T24	mean	-7.9	-2.1	-6.5	-0.9	-12.6	-9.2	-8.5	-15.7	-12.1	-18.2	-11.6	-15.9	-9.7	-16.0	-9.7
	SD	0.2	0.1	0.3	0.1	0.5	0.2	0.3	0.0	0.3	0.4	0.3	0.1	0.4	0.1	
SD-T24	2	-7.5	-2.6	-6.1	-2.4	-9.9	-9.8	-9.0	-15.7	-11.9	-18.0	-12.0	-15.3	-9.9	-16.4	
SD-T24	2	-7.9	-3.0	-6.7	-2.9	-9.3	-10.4	-9.2	-16.3	-12.4	-18.6	-12.2	-14.7	-10.1	-16.9	
SD-T24	mean	-7.7	-2.8	-6.4	-2.6	-9.6	-10.1	-9.1	-16.0	-12.1	-18.3	-12.1	-15.0	-10.0	-16.6	-9.9
	SD	0.3	0.3	0.4	0.4	0.4	0.4	0.2	0.4	0.4	0.4	0.2	0.4	0.1	0.4	
SD-T24	3	-7.8	-3.1	-6.7	-4.7	-10.8	-7.7	-8.1	-14.0	-9.2	-15.5	-9.1	-14.1	-7.6	-13.7	
SD-T24	3	-8.0	-2.5	-6.3	-4.3	-10.5	-7.1	-7.5	-13.4	-8.6	-15.3	-8.5	-14.2	-7.0	-13.7	
SD-T24	mean	-7.9	-2.8	-6.5	-4.5	-10.7	-7.4	-7.8	-13.7	-8.9	-15.4	-8.8	-14.2	-7.3	-13.7	-8.7
	SD	0.2	0.4	0.3	0.3	0.3	0.4	0.4	0.4	0.4	0.1	0.4	0.1	0.4	0.0	
SD-T24	4	-8.3	-3.4	-7.0	-5.2	-10.6	-11.7	-11.8	-15.3	-10.1	-17.1	-10.2	-13.4	-10.4	-15.7	
SD-T24	4	-8.1	-2.9	-6.5	-4.6	-11.4	-11.7	-11.3	-14.8	-10.6	-16.5	-10.3	-13.9	-10.1	-15.2	
SD-T24	mean	-8.2	-3.2	-6.7	-4.9	-11.0	-11.7	-11.6	-15.0	-10.3	-16.8	-10.2	-13.6	-10.3	-15.4	-10.1
	SD	0.1	0.4	0.4	0.4	0.5	0.0	0.4	0.4	0.3	0.4	0.1	0.4	0.2	0.3	
SD-T24	5	-13.4	-8.7	-12.0	-5.0	-13.2	-15.3	-16.1	-17.5	-13.9	-20.0	-13.0	-17.2	-15.5	-18.9	
SD-T24	5	-13.6	-8.6	-11.3	-4.6	-12.6	-15.7	-15.6	-17.7	-14.3	-19.5	-12.8	-17.8	-15.7	-19.2	
SD-T24	mean	-13.5	-8.7	-11.6	-4.8	-12.9	-15.5	-15.9	-17.6	-14.1	-19.8	-12.9	-17.5	-15.6	-19.0	-14.0
	SD	0.2	0.0	0.5	0.3	0.4	0.3	0.4	0.1	0.2	0.3	0.2	0.4	0.1	0.2	
SD-T24	6	-11.2	-4.4	-9.0	-4.4	-13.7	-14.8	-15.5	-16.7	-13.0	-19.2	-12.4	-14.6	-12.8	-17.3	
SD-T24	6	-11.2	-4.7	-8.8	-3.7	-14.7	-14.3	-15.6	-16.6	-12.6	-18.9	-12.3	-15.1	-13.1	-17.1	
SD-T24	mean	-11.2	-4.5	-8.9	-4.0	-14.2	-14.5	-15.5	-16.7	-12.8	-19.1	-12.4	-14.9	-12.9	-17.2	-12.3
	SD	0.0	0.2	0.1	0.5	0.7	0.3	0.1	0.1	0.2	0.2	0.0	0.3	0.2	0.2	
SD-T24	7	-10.4	-3.8	-7.6	-2.0	-11.2	-12.8	-12.1	-16.8	-13.0	-18.7	-13.3	-16.7	-12.6	-18.4	
SD-T24	7	-10.7	-4.3	-7.0	-2.9	-12.1	-12.2	-11.5	-16.2	-12.9	-18.2	-12.7		-12.0	-17.8	
SD-T24	mean	-10.5	-4.0	-7.3	-2.4	-11.6	-12.5	-11.8	-16.5	-12.9	-18.4	-13.0	-16.7	-12.3	-18.1	-11.3

	SD	0.2	0.3	0.4	0.6	0.6	0.4	0.4	0.4	0.1	0.4	0.4		0.4	0.4	
SD-T24	8	-5.1	-1.1	-2.4	-2.0	-9.1	-6.9	-6.4	-15.3	-10.8	-17.0	-11.1	-14.3	-8.0	-14.7	
SD-T24	8	-5.2	-1.4	-3.0	-2.3	-8.8	-6.9	-6.6	-14.8	-11.5	-17.1	-11.1	-14.8	-8.6	-15.3	
SD-T24	mean	-5.1	-1.2	-2.7	-2.2	-8.9	-6.9	-6.5	-15.1	-11.1	-17.1	-11.1	-14.6	-8.3	-15.0	-8.1
	SD	0.1	0.1	0.4	0.2	0.2	0.1	0.1	0.4	0.4	0.1	0.0	0.4	0.4	0.4	
SD-T24	9	-6.9		-3.7	-1.3	-9.7	-7.3	-7.3	-14.1	-10.0	-16.1	-10.8	-13.0	-9.0	-16.0	
SD-T24	9	-7.0	-1.7	-4.2	-1.6	-10.4	-8.0	-6.8	-14.6	-10.3	-16.7	-11.0	-13.7	-9.6	-16.7	
SD-T24	mean	-6.9	-1.7	-3.9	-1.4	-10.0	-7.7	-7.1	-14.3	-10.2	-16.4	-10.9	-13.3	-9.3	-16.3	-8.4
	SD	0.1		0.4	0.2	0.5	0.5	0.4	0.4	0.2	0.4	0.2	0.5	0.4	0.5	
SD-T24	10	-10.4	-4.0	-11.2	-4.0	-12.3	-16.2	-11.1	-16.9	-13.5	-19.3	-13.5	-16.5	-12.3	-18.3	
SD-T24	10	-10.4	-3.6	-10.6	-3.8	-11.7	-15.7	-10.6	-17.1	-13.0	-18.9	-13.0	-16.6	-11.9	-18.2	
SD-T24	mean	-10.4	-3.8	-10.9	-3.9	-12.0	-16.0	-10.8	-17.0	-13.3	-19.1	-13.2	-16.6	-12.1	-18.2	-12.1
	SD	0.0	0.3	0.4	0.1	0.5	0.3	0.4	0.2	0.4	0.3	0.3	0.1	0.3	0.1	
SI-T32	1	-7.0	-1.3	-5.2	-1.9	-12.9	-12.0	-8.7	-14.8	-12.4	-17.7	-12.2		-11.0	-16.5	
SI-T32	1	-7.3	-1.5	-5.5	-2.0	-13.8	-12.4	-9.2	-14.8	-12.4	-17.5	-11.6	-14.3	-10.8	-16.8	
SI-T32	mean	-7.2	-1.4	-5.4	-2.0	-13.3	-12.2	-8.9	-14.8	-12.4	-17.6	-11.9	-14.3	-10.9	-16.6	-9.6
	SD	0.2	0.1	0.2	0.0	0.6	0.3	0.4	0.0	0.0	0.1	0.4		0.2	0.2	
SI-T32	2	-6.7	-2.1	-5.5	-4.2	-14.9	-12.8	-8.8	-14.0	-10.4	-16.5	-10.7	-14.9	-9.0	-15.5	
SI-T32	2	-6.7	-1.4	-5.4	-4.0	-14.1	-12.9	-8.9	-14.3	-10.3	-16.5	-10.2	-15.5	-8.8	-14.8	
SI-T32	mean	-6.7	-1.7	-5.5	-4.1	-14.5	-12.9	-8.8	-14.1	-10.4	-16.5	-10.5	-15.2	-8.9	-15.2	-9.4
	SD	0.0	0.4	0.1	0.2	0.6	0.1	0.1	0.2	0.1	0.0	0.3	0.4	0.1	0.5	
SI-T32	3	-5.4		-4.5	-3.9	-14.4	-9.8	-8.4	-13.9	-9.8	-15.8	-9.6	-14.1	-8.5	-14.9	
SI-T32	3	-5.4	-0.7	-4.0	-4.6	-14.1	-9.8	-8.6	-14.0	-9.0	-16.2	-10.1	-14.2	-7.9	-14.2	
SI-T32	mean	-5.4	-0.7	-4.2	-4.2	-14.3	-9.8	-8.5	-13.9	-9.4	-16.0	-9.8	-14.1	-8.2	-14.6	-8.6
	SD	0.0		0.4	0.5	0.2	0.0	0.2	0.1	0.6	0.3	0.3	0.1	0.4	0.5	
SI-T32	4	-6.8	-2.1	-4.3	-3.2	-13.8	-11.3	-7.6	-13.8	-10.0	-16.7	-9.3	-13.2	-7.8	-14.5	
SI-T32	4	-6.4		-4.4	-3.6	-14.3	-11.1	-7.6	-13.8	-9.4	-16.7	-9.9	-13.7	-8.0	-14.8	
SI-T32	mean	-6.6	-2.1	-4.4	-3.4	-14.0	-11.2	-7.6	-13.8	-9.7	-16.7	-9.6	-13.4	-7.9	-14.7	-8.7
	SD	0.3		0.1	0.3	0.4	0.1	0.0	0.0	0.4	0.0	0.4	0.4	0.1	0.3	
SI-T32	5	-8.1	-1.8	-6.3	-1.4	-14.4	-12.5	-9.0	-15.0	-10.7	-16.5	-11.7	-13.5	-7.2	-14.9	
SI-T32	5	-7.8	-1.5	-5.9	-2.0	-13.5	-12.0	-8.5	-14.4	-10.7	-16.2	-11.4	-13.4	-7.7	-14.7	
SI-T32	mean	-7.9	-1.7	-6.1	-1.7	-13.9	-12.3	-8.7	-14.7	-10.7	-16.3	-11.5	-13.5	-7.5	-14.8	-9.2
	SD	0.2	0.2	0.2	0.4	0.6	0.4	0.3	0.4	0.0	0.3	0.2	0.1	0.4	0.2	
SI-T32	6	-8.2	-3.0	-6.0	-4.1	-10.2	-9.9	-7.2	-15.0	-10.9	-16.1	-10.6	-14.4	-8.5	-15.1	
SI-T32	6	-8.7	-2.3	-5.7	-3.4	-10.5	-10.5	-7.7	-14.9	-11.3	-16.8	-10.1	-14.4	-8.0	-14.3	
SI-T32	mean	-8.4	-2.7	-5.8	-3.7	-10.3	-10.2	-7.5	-15.0	-11.1	-16.5	-10.3	-14.4	-8.2	-14.7	-9.2
	SD	0.4	0.5	0.2	0.5	0.2	0.4	0.3	0.1	0.3	0.5	0.3	0.0	0.4	0.5	

SI-T32	7	-10.3	-2.8	-6.3	-3.2	-12.6	-12.6	-9.0	-16.0	-9.9	-17.8	-11.6	-14.1	-9.0	-15.4	
SI-T32	7	-10.0	-2.4	-6.1	-3.0	-11.8	-12.9	-8.9	-15.7	-10.7	-17.5	-11.1	-14.0	-9.0	-15.8	
SI-T32	mean	-10.2	-2.6	-6.2	-3.1	-12.2	-12.7	-9.0	-15.8	-10.3	-17.6	-11.3	-14.0	-9.0	-15.6	-9.9
	SD	0.2	0.3	0.2	0.2	0.6	0.2	0.1	0.3	0.5	0.2	0.4	0.1	0.0	0.3	
SI-T32	8	-9.8	-3.0	-6.3	-3.7	-13.0	-11.4	-9.0	-16.1	-11.0	-17.7	-11.9	-14.6	-9.2	-16.1	
SI-T32	8	-9.7	-2.4	-7.0	-3.4	-13.2	-11.8	-8.5	-15.7	-11.2	-17.7	-11.3	-15.4	-9.4	-16.4	
SI-T32	mean	-9.7	-2.7	-6.6	-3.5	-13.1	-11.6	-8.7	-15.9	-11.1	-17.7	-11.6	-15.0	-9.3	-16.2	-10.1
	SD	0.0	0.5	0.5	0.2	0.1	0.3	0.4	0.3	0.2	0.1	0.4	0.6	0.1	0.2	
SI-T32	9	-6.8	-0.2	-5.0	-2.9	-11.6	-9.6	-7.3	-15.0	-10.5	-17.2	-11.0	-15.7	-8.7	-15.7	
SI-T32	9	-6.6	-0.9	-4.7	-3.8	-12.5	-9.1	-7.6	-15.2	-10.0	-16.9	-10.7	-15.7	-8.6	-15.5	
SI-T32	mean	-6.7	-0.5	-4.9	-3.4	-12.1	-9.4	-7.4	-15.1	-10.2	-17.1	-10.8	-15.7	-8.7	-15.6	-8.9
	SD	0.1	0.5	0.2	0.6	0.7	0.3	0.2	0.1	0.4	0.3	0.2	0.0	0.1	0.1	
SI-T32	10	-8.7	-1.2	-5.3	-3.1	-12.9	-10.0	-7.7	-15.3	-11.0	-17.3	-10.9	-15.3	-9.0	-15.5	
SI-T32	10	-8.7	-0.9	-4.7	-2.2	-13.9	-10.6	-8.3	-14.9	-10.4	-17.0	-11.0	-14.9	-9.5	-15.3	
SI-T32	mean	-8.7	-1.0	-5.0	-2.7	-13.4	-10.3	-8.0	-15.1	-10.7	-17.1	-10.9	-15.1	-9.3	-15.4	-9.2
	SD	0.0	0.2	0.4	0.6	0.7	0.4	0.4	0.2	0.4	0.2	0.1	0.3	0.4	0.2	
SE-T3	1	-9.6	-2.7	-6.3	-4.1	-12.4	-12.3	-8.5	-15.8	-11.3	-17.8	-11.7	-14.3	-9.1	-16.0	
SE-T3	1	-9.6	-2.4	-6.2	-3.6	-12.1	-11.9	-7.8	-15.7	-10.8	-17.3	-11.4	-13.8	-8.8	-15.6	
SE-T3	mean	-9.6	-2.6	-6.2	-3.8	-12.2	-12.1	-8.2	-15.7	-11.1	-17.5	-11.6	-14.0	-8.9	-15.8	-9.8
	SD	0.0	0.2	0.1	0.3	0.3	0.2	0.4	0.1	0.4	0.3	0.2	0.3	0.2	0.3	
SE-T3	2	-8.8	-2.7	-6.9	-3.3	-16.4	-12.7	-8.0	-16.0	-11.5	-17.2	-11.1	-13.5	-8.7	-15.5	
SE-T3	2	-9.1	-2.6	-6.4	-3.7	-17.1	-12.3	-8.3	-16.0	-11.6	-17.4	-11.6	-13.9	-8.7	-15.3	
SE-T3	mean	-9.0	-2.6	-6.6	-3.5	-16.7	-12.5	-8.1	-16.0	-11.5	-17.3	-11.4	-13.7	-8.7	-15.4	-9.9
	SD	0.3	0.0	0.3	0.3	0.5	0.3	0.2	0.0	0.1	0.2	0.3	0.3	0.0	0.2	
SE-T3	3	-9.2	0.8	-6.8	-1.4	-15.1	-10.9	-8.9	-15.7	-11.0	-17.5	-12.5	-14.7	-9.2	-16.2	
SE-T3	3	-8.9	0.4	-6.4	-1.8	-15.6	-10.9	-8.7	-15.8	-10.6	-17.2	-12.0	-14.0	-8.9	-16.3	
SE-T3	mean	-9.1	0.6	-6.6	-1.6	-15.4	-10.9	-8.8	-15.7	-10.8	-17.3	-12.2	-14.3	-9.0	-16.3	-9.5
	SD	0.2	0.3	0.3	0.3	0.3	0.0	0.2	0.1	0.3	0.3	0.3	0.5	0.2	0.1	
SE-T3	4	-9.1	-0.1	-7.5	-4.0	-16.5	-13.7	-10.5	-17.3	-11.7	-18.3	-12.8	-13.5	-9.8	-16.7	
SE-T3	4	-9.0	-0.4	-7.5	-3.3	-15.5	-13.2	-9.9	-17.0	-12.1	-18.8	-13.2	-13.9	-10.1	-17.1	
SE-T3	mean	-9.0	-0.2	-7.5	-3.7	-16.0	-13.5	-10.2	-17.1	-11.9	-18.5	-13.0	-13.7	-10.0	-16.9	-10.6
	SD	0.1	0.2	0.0	0.5	0.7	0.4	0.4	0.2	0.3	0.4	0.3	0.3	0.2	0.3	
SE-T3	5	-8.1	0.1	-5.1	-0.6	-9.8	-7.8	-7.0	-16.0	-10.8	-18.0	-13.0	-13.9	-9.0	-16.6	
SE-T3	5	-7.9	0.2	-5.5	-0.9	-10.2	-7.4	-7.1	-16.0	-10.8	-18.0	-12.8	-14.3	-8.8	-16.9	
SE-T3	mean	-8.0	0.2	-5.3	-0.7	-10.0	-7.6	-7.1	-16.0	-10.8	-18.0	-12.9	-14.1	-8.9	-16.8	-8.9
	SD	0.2	0.1	0.3	0.2	0.3	0.3	0.1	0.0	0.0	0.1	0.2	0.2	0.1	0.2	
SE-T3	6		-1.2	-6.2	-3.4	-15.9	-9.2	-9.7	-17.9	-12.6	-19.5	-14.5	-16.4	-10.5	-18.6	

SE-T3	6	-9.3	-1.8	-6.1	-3.9	-15.5	-9.5	-9.0	-17.3	-12.0	-18.9	-14.0	-15.9	-9.8	-18.0	
SE-T3	mean	-9.3	-1.5	-6.1	-3.7	-15.7	-9.4	-9.3	-17.6	-12.3	-19.2	-14.3	-16.2	-10.1	-18.3	-10.6
	SD		0.4	0.0	0.3	0.3	0.2	0.5	0.4	0.4	0.4	0.4	0.4	0.5	0.4	
SE-T3	7	-12.8	-3.7	-9.5	-4.6	-16.6	-14.3	-10.5	-19.7	-14.6	-20.9	-15.7	-17.5	-12.6	-19.3	
SE-T3	7	-12.6	-4.2	-9.2	-5.1	-16.4	-14.0	-10.7	-19.8	-14.9	-21.3	-16.0	-18.3	-12.7	-19.5	
SE-T3	mean	-12.7	-4.0	-9.4	-4.9	-16.5	-14.2	-10.6	-19.7	-14.7	-21.1	-15.9	-17.9	-12.6	-19.4	-12.8
	SD	0.1	0.3	0.2	0.4	0.1	0.2	0.2	0.1	0.2	0.3	0.2	0.5	0.0	0.2	
SE-T3	8	-12.7	-4.7	-10.8	-7.0	-16.0	-13.7	-12.0	-20.8	-16.0	-21.8	-16.0	-19.0	-14.8	-20.0	
SE-T3	8	-12.7	-5.3	-10.3	-7.7	-15.1	-13.3	-12.3	-20.5	-16.5	-21.9	-16.0	-19.0	-14.9	-20.1	
SE-T3	mean	-12.7	-5.0	-10.5	-7.3	-15.6	-13.5	-12.2	-20.7	-16.2	-21.9	-16.0	-19.0	-14.8	-20.1	-13.9
	SD	0.0	0.4	0.3	0.5	0.7	0.3	0.2	0.2	0.3	0.1	0.0	0.0	0.0	0.0	
SE-T3	9	-14.7	-6.0	-9.9	-7.0	-16.5	-14.0	-14.4	-21.4	-16.1	-22.3	-16.4	-19.4	-14.7	-20.8	
SE-T3	9	-14.4	-5.6	-10.5	-7.8	-16.8	-14.2	-14.2	-21.0	-15.7	-22.4	-16.5	-20.0	-14.5	-20.5	
SE-T3	mean	-14.6	-5.8	-10.2	-7.4	-16.7	-14.1	-14.3	-21.2	-15.9	-22.4	-16.5	-19.7	-14.6	-20.6	-14.5
	SD	0.2	0.3	0.4	0.5	0.2	0.1	0.1	0.3	0.3	0.1	0.1	0.4	0.1	0.2	
SE-T3	10	-14.7	-7.3	-10.6	-8.5	-16.7	-14.6	-13.0	-22.0	-16.2	-22.9	-16.2	-19.3	-15.4	-21.0	
SE-T3	10	-15.0	-7.4	-10.9	-9.2		-14.4	-13.0	-22.3	-16.3	-23.3	-16.6	-20.1	-16.0	-21.3	
SE-T3	mean	-14.8	-7.4	-10.7	-8.8	-16.7	-14.5	-13.0	-22.2	-16.3	-23.1	-16.4	-19.7	-15.7	-21.2	-15.0
	SD	0.2	0.1	0.2	0.5		0.1	0.0	0.2	0.0	0.3	0.3	0.5	0.4	0.3	
SF-T4	1	-11.7	-2.9	-8.9	-4.3	-13.0	-14.3	-10.3	-17.4	-12.1	-19.7	-12.8	-17.8	-11.9	-18.0	
SF-T4	1	-11.9	-2.7	-9.0	-4.6	-14.0	-14.3	-10.1	-17.2	-12.6	-19.5	-12.8	-18.0	-11.5	-18.3	
SF-T4	mean	-11.8	-2.8	-9.0	-4.4	-13.5	-14.3	-10.2	-17.3	-12.3	-19.6	-12.8	-17.9	-11.7	-18.1	-11.8
	SD	0.1	0.2	0.1	0.2	0.7	0.0	0.1	0.1	0.3	0.1	0.0	0.2	0.2	0.2	
SF-T4	2	-9.0	-0.8	-6.6	-0.7	-10.3	-9.9	-7.5	-14.0	-10.3	-15.5	-9.2	-12.4	-7.3	-13.1	
SF-T4	2	-8.8	-0.7	-7.0	-1.5	-9.5	-10.2	-7.0	-13.2	-9.9	-15.4	-9.0	-12.3	-7.8	-13.3	
SF-T4	mean	-8.9	-0.8	-6.8	-1.1	-9.9	-10.0	-7.2	-13.6	-10.1	-15.4	-9.1	-12.4	-7.6	-13.2	-8.4
	SD	0.2	0.1	0.3	0.5	0.6	0.3	0.4	0.5	0.3	0.1	0.1	0.1	0.4	0.2	
SF-T4	3	-9.4		-7.0	-2.3	-13.6	-10.3	-9.7	-15.6	-11.8	-17.8	-12.0	-14.6	-10.1	-16.7	
SF-T4	3	-9.7	0.2	-6.9	-1.6	-13.1	-10.5	-10.1	-15.3	-11.1	-17.6	-11.7	-14.5	-9.7	-16.3	
SF-T4	mean	-9.6	0.2	-6.9	-1.9	-13.3	-10.4	-9.9	-15.5	-11.5	-17.7	-11.8	-14.6	-9.9	-16.5	-9.8
	SD	0.2		0.0	0.5	0.4	0.1	0.3	0.2	0.4	0.2	0.2	0.0	0.3	0.3	
SF-T4	4	-9.4	-3.4	-8.1	-2.3	-13.5	-10.0	-10.2	-15.6	-12.2	-18.2	-11.6	-14.1	-10.7	-16.6	
SF-T4	4	-9.7	-3.2	-7.7	-2.0	-14.1	-10.5	-10.5	-15.4	-11.6	-17.7	-11.1	-14.2	-10.7	-16.5	
SF-T4	mean	-9.5	-3.3	-7.9	-2.1	-13.8	-10.2	-10.4	-15.5	-11.9	-17.9	-11.4	-14.2	-10.7	-16.6	-10.5
	SD	0.2	0.1	0.3	0.2	0.4	0.4	0.2	0.2	0.4	0.3	0.3	0.1	0.0	0.1	
SF-T4	5	-10.4	-3.4	-7.8	-3.5	-16.6	-12.0	-11.9	-15.6	-10.8	-17.8	-11.9	-14.1	-11.0	-16.6	
SF-T4	5	-10.8	-3.2	-8.2	-3.7	-15.6	-12.7	-12.2	-16.0	-11.4	-18.1	-12.3	-14.4	-11.5	-17.0	

SF-T4	mean	-10.6	-3.3	-8.0	-3.6	-16.1	-12.3	-12.0	-15.8	-11.1	-18.0	-12.1	-14.3	-11.3	-16.8	-11.1
	SD	0.3	0.1	0.3	0.1	0.7	0.5	0.3	0.3	0.4	0.2	0.3	0.2	0.3	0.3	
SF-T4	6	-7.1	-1.8	-4.1	-1.6	-15.6	-9.1	-8.7	-14.4	-9.1	-17.0	-10.3	-11.7	-7.4	-14.3	
SF-T4	6	-6.7	-2.6	-4.7	-0.7	-14.7	-9.5	-8.0	-14.5	-9.4	-16.8	-10.3	-12.2	-7.6	-14.6	
SF-T4	mean	-6.9	-2.2	-4.4	-1.1	-15.2	-9.3	-8.3	-14.4	-9.3	-16.9	-10.3	-12.0	-7.5	-14.4	-8.5
	SD	0.3	0.5	0.4	0.6	0.7	0.3	0.5	0.1	0.2	0.2	0.0	0.3	0.1	0.2	
SF-T4	7	-10.4	-2.3	-9.3	-2.7	-14.2	-15.0	-10.0	-16.7	-12.0	-18.9	-12.0	-16.5	-11.6	-16.9	
SF-T4	7	-10.0	-2.2	-8.6	-2.0	-14.8	-14.3	-10.3	-16.6	-12.2	-18.5	-11.8	-15.7	-11.7	-17.4	
SF-T4	mean	-10.2	-2.2	-9.0	-2.4	-14.5	-14.7	-10.1	-16.7	-12.1	-18.7	-11.9	-16.1	-11.7	-17.1	-11.2
	SD	0.3	0.1	0.5	0.5	0.4	0.5	0.2	0.1	0.2	0.2	0.1	0.5	0.1	0.3	
SF-T4	8	-6.9		-5.7	-0.3	-10.2	-11.3	-8.1	-14.0	-10.3	-17.0	-10.2	-13.7	-9.1	-15.1	
SF-T4	8	-7.2	0.9	-5.5	-0.5	-9.5	-10.8	-7.6	-14.4	-9.8	-17.0	-9.6	-12.9	-8.7	-15.4	
SF-T4	mean	-7.1	0.9	-5.6	-0.4	-9.8	-11.0	-7.8	-14.2	-10.1	-17.0	-9.9	-13.3	-8.9	-15.3	-8.5
	SD	0.2		0.2	0.1	0.5	0.3	0.3	0.3	0.3	0.0	0.4	0.5	0.3	0.2	
SF-T4	9	-7.7	0.8	-5.2	-0.4	-11.8	-10.7	-7.8	-14.6	-11.8	-17.6	-10.7	-13.8	-9.3	-16.4	
SF-T4	9	-7.1	0.7	-4.9	-1.1	-10.8	-11.0	-7.3	-14.4	-11.4	-17.3	-10.8	-14.0	-9.7	-16.2	
SF-T4	mean	-7.4	0.8	-5.0	-0.8	-11.3	-10.8	-7.6	-14.5	-11.6	-17.5	-10.7	-13.9	-9.5	-16.3	-8.8
	SD	0.4	0.1	0.2	0.4	0.7	0.2	0.3	0.1	0.3	0.2	0.1	0.1	0.3	0.1	
SF-T4	10	-7.0	-1.9	-3.0	-1.7	-9.1	-6.7	-6.2	-13.0	-9.7	-15.9	-11.8	-14.0	-7.9	-14.6	
SF-T4	10	-6.6	-2.3	-3.5	-2.2	-9.8	-7.2	-6.6	-13.6	-9.5	-16.5	-11.4	-13.9	-8.4	-15.1	
SF-T4	mean	-6.8	-2.1	-3.3	-2.0	-9.5	-7.0	-6.4	-13.3	-9.6	-16.2	-11.6	-14.0	-8.2	-14.8	-8.0
	SD	0.3	0.3	0.4	0.3	0.5	0.3	0.2	0.4	0.1	0.4	0.2	0.1	0.3	0.4	
SI-T3	1	-8.4	-0.9	-4.6	-0.6	-11.8	-9.8	-8.2	-13.1	-8.7	-15.9	-10.9	-14.4	-8.5	-14.8	
SI-T3	1	-9.1	-0.7	-4.5	-0.9	-10.8	-10.0	-8.1	-14.0	-9.6	-16.8	-11.2	-15.0	-9.4	-15.4	
SI-T3	mean	-8.7	-0.8	-4.5	-0.8	-11.3	-9.9	-8.1	-13.6	-9.1	-16.3	-11.1	-14.7	-8.9	-15.1	-8.6
	SD	0.5	0.1	0.1	0.2	0.7	0.2	0.1	0.6	0.6	0.6	0.2	0.4	0.6	0.4	
SI-T3	2	-8.9	-1.6	-5.8	-0.9	-11.4	-11.4	-9.1	-14.3	-9.9	-16.7	-11.7	-14.2	-9.5	-15.9	
SI-T3	2	-9.5	-0.9	-5.5	-1.1	-10.7	-10.9	-9.1	-14.7	-10.3	-17.0	-12.0	-14.5	-9.9	-16.5	
SI-T3	mean	-9.2	-1.2	-5.6	-1.0	-11.1	-11.2	-9.1	-14.5	-10.1	-16.9	-11.8	-14.3	-9.7	-16.2	-9.3
	SD	0.4	0.5	0.2	0.1	0.5	0.3	0.0	0.3	0.3	0.2	0.2	0.2	0.3	0.4	
SI-T3	3	-9.0	-0.1	-6.6	-1.7	-12.9	-10.4	-9.1	-15.0	-11.2	-17.5	-11.9	-14.7	-10.0	-16.5	
SI-T3	3	-9.4	-0.2	-6.3	-1.6	-11.8	-10.5	-9.1	-15.3	-11.4	-17.8	-12.3	-14.7	-9.9	-16.8	
SI-T3	mean	-9.2	-0.2	-6.5	-1.6	-12.4	-10.5	-9.1	-15.2	-11.3	-17.6	-12.1	-14.7	-10.0	-16.7	-9.6
	SD	0.3	0.1	0.2	0.1	0.7	0.1	0.0	0.2	0.1	0.2	0.3	0.0	0.1	0.2	
SI-T3	4	-8.6	-0.4	-5.2	-0.8	-12.6	-11.8	-8.7	-14.7	-11.4	-17.4	-11.7	-15.5	-9.5	-15.9	
SI-T3	4	-8.8	-0.1	-5.2	-1.5	-11.6	-11.5	-8.7	-15.1	-10.9	-17.7	-12.0	-15.2	-9.8	-16.4	
SI-T3	mean	-8.7	-0.2	-5.2	-1.2	-12.1	-11.6	-8.7	-14.9	-11.1	-17.5	-11.8	-15.4	-9.6	-16.2	-9.3

	SD	0.2	0.2	0.0	0.5	0.7	0.2	0.0	0.3	0.4	0.2	0.2	0.2	0.3	0.3	
SI-T3	5	-10.8	-3.9	-9.3	-2.8	-13.0	-12.7	-9.7	-15.8	-11.6	-18.0	-12.1	-14.7	-10.3	-16.7	
SI-T3	5	-11.1	-3.8	-8.9	-3.2	-13.7	-12.7	-9.9	-15.7	-12.2	-18.4	-12.7	-15.3	-10.8	-17.3	
SI-T3	mean	-11.0	-3.8	-9.1	-3.0	-13.3	-12.7	-9.8	-15.7	-11.9	-18.2	-12.4	-15.0	-10.6	-17.0	-11.0
	SD	0.2	0.0	0.2	0.3	0.5	0.0	0.1	0.0	0.4	0.2	0.4	0.4	0.3	0.4	
SI-T3	6	-11.7	-2.2	-9.0	-2.6	-13.5	-13.9	-13.0	-16.3	-11.8	-18.3	-13.1	-15.1	-10.8	-16.7	
SI-T3	6	-12.1	-2.9	-8.7	-2.6	-12.5	-13.9	-13.1	-16.4	-11.6	-18.7	-12.5	-15.1	-11.4	-17.0	
SI-T3	mean	-11.9	-2.5	-8.8	-2.6	-13.0	-13.9	-13.0	-16.3	-11.7	-18.5	-12.8	-15.1	-11.1	-16.8	-11.4
	SD	0.3	0.4	0.3	0.0	0.7	0.0	0.1	0.1	0.1	0.3	0.4	0.0	0.4	0.3	
SI-T3	7	-10.8	-2.1	-7.7	-2.4	-11.3	-11.7	-11.7	-15.4	-11.4	-17.6	-12.7	-15.3	-11.3	-16.3	
SI-T3	7	-10.8	-2.1	-8.1	-2.1	-11.0	-12.3	-12.3	-15.4	-12.0	-18.2	-13.1	-15.4	-11.4	-16.7	
SI-T3	mean	-10.8	-2.1	-7.9	-2.2	-11.2	-12.0	-12.0	-15.4	-11.7	-17.9	-12.9	-15.3	-11.3	-16.5	-10.7
	SD	0.0	0.0	0.3	0.2	0.2	0.5	0.4	0.0	0.4	0.4	0.3	0.1	0.1	0.3	
SI-T3	8	-9.4	-1.1	-7.2	-1.0	-11.3	-11.6	-11.7	-15.0	-11.7	-17.7	-11.8	-14.2	-10.3	-16.6	
SI-T3	8	-9.5	-0.7	-7.6	-1.7	-10.4	-12.0	-12.0	-15.0	-11.6	-18.0	-11.3	-14.6	-9.7	-16.5	
SI-T3	mean	-9.5	-0.9	-7.4	-1.3	-10.8	-11.8	-11.8	-15.0	-11.6	-17.9	-11.6	-14.4	-10.0	-16.6	-10.1
	SD	0.1	0.3	0.3	0.5	0.6	0.3	0.2	0.0	0.1	0.2	0.3	0.3	0.4	0.1	
SI-T3	9	-9.2	-2.4	-6.9	-1.1	-11.5	-12.5	-9.5	-14.7	-10.0	-17.3	-11.5	-15.2	-9.4	-15.9	
SI-T3	9	-9.7	-2.1	-6.5	-1.3	-12.6	-12.9	-9.9	-14.5	-10.2	-17.6	-12.1	-15.2	-10.0	-16.3	
SI-T3	mean	-9.4	-2.3	-6.7	-1.2	-12.1	-12.7	-9.7	-14.6	-10.1	-17.5	-11.8	-15.2	-9.7	-16.1	-9.8
	SD	0.4	0.2	0.3	0.1	0.7	0.3	0.3	0.2	0.2	0.2	0.4	0.0	0.4	0.2	
SI-T3	10	-8.6	-2.2	-5.9	-1.3	-11.9	-10.5	-8.5	-15.1	-11.1	-17.5	-12.5	-15.0	-9.7	-16.2	
SI-T3	10	-8.4	-1.5	-6.1	-1.2	-11.1	-10.9	-8.7	-15.2	-11.2	-17.8	-12.4	-15.3	-10.0	-16.6	
SI-T3	mean	-8.5	-1.9	-6.0	-1.2	-11.5	-10.7	-8.6	-15.2	-11.1	-17.6	-12.4	-15.1	-9.8	-16.4	-9.6
	SD	0.1	0.5	0.1	0.1	0.6	0.3	0.1	0.1	0.1	0.2	0.1	0.2	0.2	0.4	

Table C.2. Summary of mean within-individual amino acid $\delta^{13}\text{C}$ (‰) values for hair keratin.

Individual	Essential Amino Acids						Non-Essential Amino Acids									MB*
	Phe	Val	Leu	Ile	Lys	Thr	Ala	Ser	Gly	Asx	Glx	Pro	Arg	Tyr		
SI-T74																
Mean	-19.3	-16.8	-19.1	-12.7	-13.6	-3.6	-11.8	-2.6	-10.4	-9.2	-7.2	-12.1	-12.8	-18.1	-11.4	
SD	1.5	0.8	1.0	1.3	1.4	0.9	1.6	1.7	1.5	1.1	1.8	2.2	1.3	1.0	1.0	

Range	4.3	2.8	3.2	4.2	4.4	3.2	6.1	4.3	4.3	3.7	6.3	6.8	4.2	3.2	3.3
Min	-21.3	-18.5	-20.4	-14.5	-16.0	-5.5	-14.1	-5.0	-12.9	-10.4	-10.1	-15.2	-14.7	-19.9	-12.5
Max	-17.0	-15.7	-17.2	-10.3	-11.6	-2.3	-8.0	-0.7	-8.6	-6.7	-3.8	-8.4	-10.5	-16.7	-9.2
SD-T24															
Mean	-16.6	-15.8	-17.9	-11.8	-11.6	-3.2	-11.2	-3.5	-11.4	-8.9	-7.1	-10.5	-10.8	-15.2	-10.5
SD	1.6	1.2	1.4	1.6	1.4	1.4	3.4	2.1	1.6	2.4	2.8	3.3	2.5	1.4	1.9
Range	5.3	3.9	4.4	5.2	4.4	4.0	9.1	7.5	5.3	8.4	8.9	9.4	8.3	4.2	5.9
Min	-19.0	-17.6	-19.8	-14.1	-13.2	-4.9	-16.0	-8.7	-14.2	-13.5	-11.6	-15.9	-15.6	-17.5	-14.0
Max	-13.7	-13.7	-15.4	-8.9	-8.8	-0.9	-6.9	-1.2	-8.9	-5.1	-2.7	-6.5	-7.3	-13.3	-8.1
SI-T32															
Mean	-15.3	-14.8	-16.9	-10.6	-10.8	-3.2	-11.3	-1.7	-13.1	-7.8	-5.4	-8.3	-8.8	-14.5	-9.3
SD	0.7	0.7	0.6	0.8	0.8	0.8	1.3	0.8	1.3	1.5	0.8	0.6	1.0	0.8	0.5
Range	2.0	2.1	1.7	3.0	2.3	2.5	3.5	2.2	4.2	4.8	2.4	1.6	3.4	2.3	1.5
Min	-16.6	-15.9	-17.7	-12.4	-11.9	-4.2	-12.9	-2.7	-14.5	-10.2	-6.6	-9.0	-10.9	-15.7	-10.1
Max	-14.6	-13.8	-16.0	-9.4	-9.6	-1.7	-9.4	-0.5	-10.3	-5.4	-4.2	-7.4	-7.5	-13.4	-8.6
SE-T3															
Mean	-18.1	-18.2	-19.6	-13.2	-14.0	-4.5	-12.2	-2.8	-15.2	-10.9	-7.9	-10.2	-11.3	-16.2	-11.6
SD	2.1	2.5	2.3	2.3	2.0	2.6	2.3	2.7	2.2	2.5	2.1	2.3	2.8	2.6	2.3
Range	5.8	6.5	5.8	5.5	5.1	8.1	6.9	8.0	6.7	6.8	5.4	7.2	7.0	6.0	6.1
Min	-21.2	-22.2	-23.1	-16.3	-16.5	-8.8	-14.5	-7.4	-16.7	-14.8	-10.7	-14.3	-15.7	-19.7	-15.0
Max	-15.4	-15.7	-17.3	-10.8	-11.4	-0.7	-7.6	0.6	-10.0	-8.0	-5.3	-7.1	-8.7	-13.7	-8.9
SF-T4															
Mean	-15.9	-15.1	-17.5	-11.0	-11.2	-2.0	-11.0	-1.5	-12.7	-8.9	-6.6	-9.0	-9.7	-14.3	-9.7
SD	1.5	1.3	1.2	1.1	1.1	1.3	2.3	1.6	2.4	1.8	2.0	1.8	1.6	1.7	1.4
Range	4.9	4.0	4.2	3.0	3.7	4.0	7.7	4.2	6.6	5.0	5.7	5.6	4.2	5.9	3.8
Min	-18.1	-17.3	-19.6	-12.3	-12.8	-4.4	-14.7	-3.3	-16.1	-11.8	-9.0	-12.0	-11.7	-17.9	-11.8
Max	-13.2	-13.3	-15.4	-9.3	-9.1	-0.4	-7.0	0.9	-9.5	-6.8	-3.3	-6.4	-7.5	-12.0	-8.0
SI-T3															
Mean	-16.4	-15.0	-17.6	-11.0	-12.1	-1.6	-11.7	-1.6	-11.9	-9.7	-6.8	-10.0	-10.1	-14.9	-9.9
SD	0.5	0.7	0.6	0.9	0.6	0.7	1.2	1.1	0.8	1.1	1.5	1.7	0.7	0.4	0.9
Range	1.9	2.7	2.2	2.8	1.8	2.2	4.0	3.6	2.5	3.4	4.6	4.9	2.4	1.1	2.8
Min	-17.0	-16.3	-18.5	-11.9	-12.9	-3.0	-13.9	-3.8	-13.3	-11.9	-9.1	-13.0	-11.3	-15.4	-11.4
Max	-15.1	-13.6	-16.3	-9.1	-11.1	-0.8	-9.9	-0.2	-10.8	-8.5	-4.5	-8.1	-8.9	-14.3	-8.6

*MB indicates calculated $\delta^{13}\text{C}$ mass balance values.