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PAIRED RADIOCARBON DATING ON HUMAN SAMPLES AND CAMELID FIBERS AND TEXTILES FROM NORTHERN CHILE: THE CASE OF PICA 8 (TARAPACÁ)

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ABSTRACT. Pica 8 is a Late Intermediate Period (AD 900–1450) cemetery located in the Atacama Desert. Burials at the site present unexpectedly high variability in $\delta^{13}C$ (–8‰ to –16‰) and $\delta^{15}N$ (10‰ to 24‰) values in their skeletal tissues, implying highly diverse diets. There are two possible explanations for this variability: the first is diachronic change in diet while the second involves synchronic sociocultural distinctions. To distinguish between them a radiocarbon (^{14}C) dating program (n = 23) was initiated. The presumed importance of marine foods adds the complication of a marine reservoir effect. To address this problem, paired ^{14}C dates were obtained on human bone and camelid textiles from nine graves. The results fall into two groups, one showing an average offset of 117 ± 9 ^{14}C yr, and the other no statistically significant offsets. We conclude that the contribution of marine foods to bone collagen at Pica 8 was less than previously supposed. Other factors must be invoked to account for the unusually high human $\delta^{15}N$ values at the site. Manuring crops with sea-bird guano emerges as a probable explanation. No relationship with chronology is seen implying the presence of considerable diversity in diets and hence lifeways within the Pica 8 community.

KEYWORDS: Atacama Desert, marine reservoir effect, stable carbon and nitrogen isotopes.

INTRODUCTION

A robust chronology is essential for the interpretation of patterns found in the archaeological record and, by implication, the social processes underlying them. In this paper, we provide an example focused on the cemetery of Pica 8, one of a group of oasis sites on the edges of the Atacama Desert, northern Chile (Figure 1). The site belongs to the Pica-Tarapacá Cultural complex of the Late Intermediate Period (LIP), AD 900–1450 (Núñez 1984; Uribe 2006). This complex extends from the Camiña River to the north, the Loa River to the south, and from the Pacific coast to the west to the Andean pre-cordillera and highlands to the east (Uribe 2006). Even though Pica 8 is located approximately 90 km from the coast, the archaeological evidence (fish and shellfish remains, manta ray eggs, marine bird feathers) suggests connections between the oases and the seaside (Núñez 1984; Zlatar 1984; Moragas 1995; Briones et al. 2005; Uribe 2006).

A total of 254 burials were excavated from the cemetery (Núñez 1965, 1984). Thus far, a total of 82 skeletons have been analyzed for sex and age estimations as well as paleopathology and violence-related trauma (Retamal et al. 2012; Pacheco and Retamal 2017). Stable isotope analysis on 30 human skeletons revealed a surprising degree of dietary variability, with some individuals interpreted as consuming significant amounts of marine foods (despite the site's location approximately 90 km inland), while the diets of other individuals appeared entirely terrestrial (Santana-Sagredo et al. 2015a). Moreover, the graves of individuals exhibiting these different diets appeared to be grouped spatially into different sectors of the cemetery. A question then arose regarding the extent to which the observed isotopic-spatial pattern was diachronic or "synchronic" (see below). The available chronology for the site has relied mainly on pottery typology, which lacks sufficient resolution in this region to confidently distinguish

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Figure 1 Map of northern Chile. The cemetery of Pica 8 is indicated by an arrow. Map slightly modified from Santana-Sagredo et al. (2015a).

between these alternatives, although lending more support to the second scenario of considerable dietary heterogeneity within the community using Pica 8 for burial purposes. Here, we report the results of a radiocarbon dating program (n = 23) focused on this question.

The isotopic results obtained for the Pica 8 human remains revealed the existence of considerable variability in collagen and apatite $\delta^{13}C$, and in collagen $\delta^{15}N$ values (Santana-Sagredo et al. 2015a). At least three dietary groupings could be identified in Pica 8. The first exhibits high $\delta^{13}C$ values for bone collagen and apatite together with high $\delta^{15}N$ values, suggesting a marine-C_4 (maize) based diet. The second grouping of high $\delta^{13}C$ but lower $\delta^{15}N$ values (although still relatively high at 14–15‰) suggests a focus on maize with a lower reliance on marine resources. Finally, the third grouping suggests a terrestrial-C_3 based diet with the lowest values for both carbon and nitrogen isotopes. Some of this variability can be related to the sectors—nine in total, designated A through J (Núñez 1984; Zlatar 1984)—in which the burials were found. In our previous study, individuals from sectors A, B, D, E, F, G, and I were analyzed (Santana-Sagredo et al. 2015a). Sector I contains mainly individuals with very high $\delta^{15}N$ values of more than 20‰, while most individuals in Sectors A and G exhibit lower values of around 14–15‰ (Figure 2). Other sectors, such as D, are more variable, containing individuals with both high and low $\delta^{15}N$ values.

While the cemetery can be confidently placed in the LIP, this period spans at least 500 yr, and it is possible that the observed isotopic variability is related to changing subsistence practices over time. This change could be due to increased contacts with the coast (clearly attested by finds of desiccated marine fish and shellfish, manta ray eggs, and marine bird feathers), or to intensification of farming practices based on manuring of crop staples [as suggested by experimental studies on modern crops with seabird guano by Szpak et al. (2012a, 2012b)]. The alternative is that the community at Pica 8 comprised a cosmopolitan mix of groups of people with different origins and diets, many of whom maintained their distinct cuisines. Resolving the question of

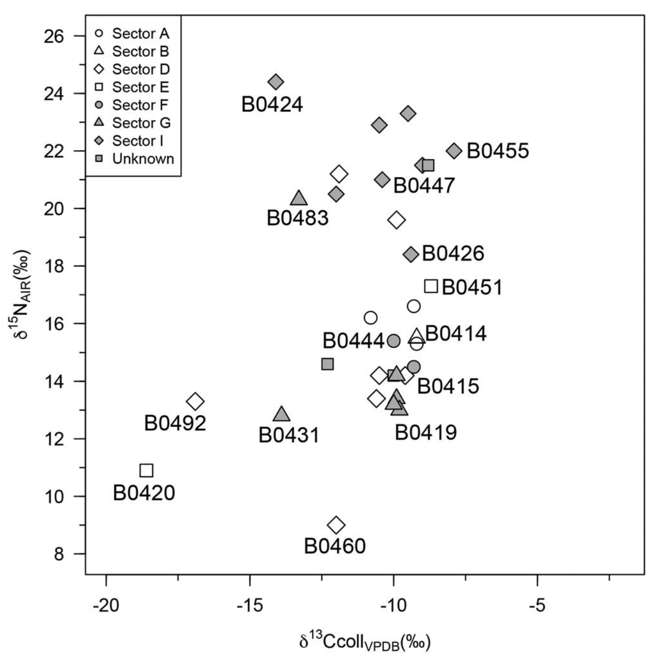

Figure 2 Bivariate plot showing $\delta^{13}C$ and $\delta^{15}N$ values from bone collagen for Pica 8, identified by sector (Santana-Sagredo et al. 2015a). Individuals selected for radiocarbon dating are indicated by their inventory numbers.

which explanation is more likely will greatly improve our understanding of the Pica-Tarapacá Complex, and could (assuming the "synchronic" view is supported) provide new avenues of research into the conditions under which such "sustained multiculturalism" arose, and how and why it was maintained over multiple generations, if not centuries.

Central to choosing between the two alternatives set out above is a robust chronology. What prevents this from being a straightforward task is the complication of the inferred high marine contribution to the diets of those individuals with elevated $\delta^{15}N$ values, above 18‰ (Santana-Sagredo et al. 2015a). Moreover, the marine reservoir effect (MRE) is known to be extremely complex off the west coast of South America, being heavily influenced by deep-water upwelling, the intensity of which varies spatially and temporally, and by shifts in oceanic circulation due to influences such as the El Niño Southern Oscillation (ENSO) (Owen 2002; Fontugne et al. 2004; Ortlieb et al. 2011; Latorre et al. 2015).

The main aim of this study is to resolve the chronology of the Pica 8 cemetery and, in particular, whether the observed isotopic/dietary variability is diachronic or "synchronic." The term "synchronic" in this context is not meant to imply that all the burials are necessarily exactly contemporary, but rather that the observed isotopic variation bears no relationship with chronology. The underlying inference, however, is that the rejection of the diachronic explanation implies isotopic/dietary heterogeneity within a single "living" community. To achieve this, 9 paired ^{14}C determinations were undertaken on human bone and camelid textiles in order to address the marine reservoir offset for this region and period. We expect human collagen to be most affected by the MRE, whereas the camelid textiles should be entirely unaffected. In addition to the paired dates, 5 additional burials were selected for ^{14}C dating. No textiles or other appropriate terrestrial dating materials could be associated with these individuals, but they were included in order to provide more comprehensive spatial coverage of the various sectors of the Pica 8 cemetery, which previous research suggested may have been a factor in the observed isotopic/dietary variability (Santana-Sagredo et al. 2015a).

Archaeological and Chronological Context of the Pica-Tarapaca Cultural Complex

The Late Intermediate Period (AD 900–1450) in northern Chile falls between the Tiwanaku State (preceding) and Inca (following) Empire. However, the Tarapacá region was not within the Tiwanaku sphere of influence during the Middle Horizon, AD 400–900 (Berenguer and Dauelsberg 1989). Instead, the LIP here was preceded by an extended Formative Period, from approximately 1000 BC to AD 900. The LIP populations were direct heirs of the Formative Period groups that inhabited the Tarapacá lowlands. During the LIP these populations expanded their occupation to the highlands. The LIP lacked overarching sociopolitical structures, being characterized instead by segmented and autonomous communities (Uribe 2006). In order to provide the economic necessities, increased mobility and trade occurred between these communities, thus connecting highland, inland, and coastal zones (Schiappacasse et al. 1989). However, it is still not clear how and at what scale human mobility occurred. LIP social organization, settlement and mobility have conventionally been interpreted following models that are strongly influenced by ethnohistorical accounts involving the imposition of colonies from the highlands to the valleys and coast (Murra 1972) or the exchange of goods controlled by llama caravans from the highlands (Núñez and Dillehay 1995). There is an ongoing debate on this matter.

Much of the current LIP chronological framework for the Pica-Tarapacá Complex is based on ceramic seriation, supported by thermoluminescence (TL) determinations (Uribe et al. 2007).

The main pottery type that characterizes this cultural complex is known as Pica Charcollo (PCH) with an age range of AD 950–1470, also including variants such as Pica Chiza (PCHZ) and Pica Gris Alisado (PGA). Only six ^{14}C dates on archaeological material have been published so far for the interior part of the Tarapacá region, specifically from the Pica 8 cemetery itself (Núñez 1976). However, two of these (on maize cobs) give calibrated dates post-AD 1500, too recent for what is clearly a pre-Columbian cemetery; they likely represent intrusive material. A third date on a camelid textile from tomb 6 shows a determination between cal AD 968–1270 (IVIC-173: 930 ± 90 BP). The final three determinations are on human bone collagen, specifically individuals SDT11 (Beta 220922: 1050 ± 40 BP: AD 900–1030), B0447 (Beta-220923: 900 ± 40 BP: AD 1030–1230), and B0438 (Beta-220924: 810 ± 40 BP: AD 1170–1280) (Uribe et al. 2007). All four would thus appear to fall within the LIP. However, since there is evidence of marine dietary input at Pica 8 (Santana-Sagredo et al. 2015a), these dates may be subject to the MRE. No δ^{15}N measurements are associated with these dates, limiting their usefulness. Overall, given the paucity of absolute dates, our results will contribute to building a more robust chronological framework for the LIP in northern Chile.

Marine Reservoir Effect in Northern Chile

The marine reservoir effect (MRE) is defined as the difference in the ^{14}C content present in terrestrial and marine organisms, with the latter being depleted in ^{14}C (Stuiver et al. 1986; Stuiver and Brazuinas 1993). In contrast to terrestrial environments and organisms, the CO_2 present in the ocean is not in equilibrium with the atmosphere, being influenced by sources of "old" organic and inorganic carbon. Thus, dates obtained for modern marine organisms will be significantly older compared to those from the terrestrial biome. This average global ocean MRE difference has been estimated at around 400 yr (Stuiver et al. 1986; Stuiver and Brazuinas 1993), but local deviations from this can vary significantly depending on upwelling intensity and latitude. Inputs from freshwater systems can have their own reservoir offsets (Fernandes et al. 2014; Schulting et al. 2014) but this is not a factor in northern Chile, which has no significant watercourses. This additional local variation, designated ΔR, needs to be considered when correcting the radiocarbon dates for any MRE in addition to the global R average (Stuiver et al. 1986; Stuiver and Brazuinas 1993).

Northern Chile and southern Peru are characterized by a strong upwelling system (Ortlieb et al. 2011; Latorre et al. 2015). The Humboldt Current is characterized by high productivity and biodiversity, due to the high nutrient content of upwelled waters. Upwelling also brings dissolved inorganic carbon depleted in ^{14}C from the ocean bottom to the surface (Latorre et al. 2015). There are relatively limited data pertaining to the region's ΔR values: the ^{14}CHRONO Marine Reservoir Database (http://calib.qub.ac.uk/marine/) shows seven data points for northern Chile and southern Peru, with modern local ΔR values ranging from less than 50 yr up to almost 400 yr. Most of the information available in the database comes from Ortlieb et al. (2011), who provide the most complete study on the MRE for northern Chile and southern Peru. Using a total of 46 paired dates on marine and terrestrial samples their data show considerable variability in ΔR through time. During the early Holocene ΔR values average 511 ± 278 yr, decreasing significantly during the middle Holocene to 226 ± 98 yr, before increasing again in the late Holocene to 355 ± 105 yr. The authors relate this variability to changes in upwelling and suggest a stronger influence of the ENSO during the late Holocene.

A recent study by Latorre et al. (2015) on archaeological shell middens from Caleta Vitor, northern Chile, shows ΔR values somewhat higher than those obtained by Ortlieb et al. (2011)

during the early and mid-Holocene, emphasizing the difficulties involved in estimating ΔR. Compounding this is the spatial as well as temporal variation. The upwelling system for northern Chile shows significant differences compared to central Chile. Carré et al. (2016) provide an average ΔR value of 168 ± 69 yr for the late Holocene in central Chile, compared to 355 ± 105 yr for northern Chile. The difference between the early Holocene of central and northern Chile is even higher, with ΔR values of 31 ± 156 yr and 528 ± 301 yr, respectively (Latorre et al. 2015; Carré et al. 2016). Caution needs to be taken when interpreting the radiocarbon dates possibly affected by the MRE, considering the high variation in the ΔR values along the Chilean coast.

MATERIALS AND METHODS

A total of 23 samples from Pica 8 were selected for ^{14}C dating, comprising adult rib samples and one hair sample from 13 distinct individuals, 9 of which could be paired with associated textiles to address the MRE (Tables 1 and 2). Adults were chosen to avoid complications in the interpretation of δ^{15}N values due to nursing effects. We were able to obtain a new bone sample from only one of the three individuals (B0447) previously dated by Uribe et al. (2007).

The burials at Pica 8 were found in a naturally mummified state with the bodies completely preserved. All the soft tissues were removed in the 1970s to facilitate osteological analysis (Aspillaga personal communication 2016). The human bone and hair samples reported here were previously analyzed for $\delta^{13}C_{coll}$ and δ^{15}N, and $\delta^{13}C_{ap}$ (as appropriate). Approximately one-third are argued to show considerable consumption of marine resources, while others suggest a mixed marine and terrestrial diet, and a few others a largely terrestrial diet (Figure 2; Santana-Sagredo et al. 2015a).

Nine camelid textile and fiber samples were selected to pair with human bone. These were all directly associated with the bodies, comprising tunics and wrappings for the deceased, a textile bag, a loincloth, a camelid hair bundle, and one example of a camelid "hairball" found inside the mouth of one individual [the practice of leaving hairballs inside the mouths of the deceased has been documented at a Formative Period coastal site at Pisagua (Agüero personal communication); Figure 3].

Given the excellent preservation at the site due to the extremely arid conditions of the Atacama, neither the bone nor textile samples had been treated with any consolidants or conservation materials. All the analyses were carried out at the Oxford Radiocarbon Accelerator Unit (ORAU), University of Oxford. Bone collagen samples, as well has hair and textile (camelid fibers) samples were pretreated following the protocols of Brock et al. (2010). The human bone collagen samples as well as the hair keratin sample had been previously prepared for stable isotope analysis (Santana-Sagredo et al. 2015a) at the Research Laboratory for Archaeology and the History of Art (RLAHA), University of Oxford.

Two out of the four new bone collagen samples, together with three of those previously prepared in RLAHA, presented high C/N ratios above 3.45 and were considered unacceptable for dating following the stricter ORAU standards rather than DeNiro's (1985) widely accepted range of 2.9 to 3.6 for dietary stable isotope research. Since the samples were very well preserved, the slight elevation in their C/N ratios may have been due to the presence of traces of lipids. Therefore, two solvent washes with acetone, methanol, and chloroform were performed on these five samples. The resultant C/N ratios decreased from >3.45 to between 3.1 and 3.2, so that all samples met the quality control criteria.

Table 1 Carbon and nitrogen stable isotope values and radiocarbon years for human and textile paired samples.

Sample	Inventory	Sector	Lab code OxA-	$\delta^{13}C_{coll}$	$\delta^{15}N$	$\delta^{13}C_{ap}$	^{14}C yr	±	^{14}C offset	cal AD	R_combine
Bone (rib)	B0414	B	33425	–9.2	15.5	–3.6	1035	27	127	993–1147	T = 8.8 (5% 3.8)
Textile (tunic)	B0414/A03827	B	32817	–14.2	13.0	—	908	33	127	1047–1266	—
Bone (rib)	B0415	D	33426	–9.6	14.2	–3.9	896	30	–4	1070–1266	T = 0.0 (5% 3.8)
Textile (bag?)	B0415*	D	32818	–17.5	8.6	—	900	32	–4	1051–1266	—
Human hair	B0460	D	32728	–12.0	9.0	—	935	24	25	1046–1217	T = 0.4 (5% 3.8)
Textile (tunic)	B0460/A03829	D	32819	–16.2	9.6	—	910	32	25	1046–1264	—
Bone (rib)	B0492	D	33429	–16.9	13.3	–11.7	980	28	26	1028–1163	T = 0.5 (5% 3.8)
Textile (bag)	B0492/A03830	D	32882	–19.4	9.9	—	954	26	26	1039–1191	—
Bone (rib)	B0420	E	V-2645-51	–18.6	10.9	–13.0	744	24	110	1271–1383	T = 7.5 (5% 3.8)
Textile (bag?)	B0420*	E	32820	–12.8	16.5	—	634	32	110	1301–1414	—
Bone (rib)	B0451	E	V-2648-36	–8.7	17.3	–3.9	948	25	–274	1045–1207	—
Textile (hairball)	B0451/A03841	E	32972	–15.8	15.2	—	1222	31	–274	769–969	—
Bone (rib)	B0444	F	33427	–10.0	15.4	–4.6	978	28	109	1029–1173	T = 6.3 (5% 3.8)
Textile (tunic)	B0444*	F	32821	–16.3	9.3	—	869	33	109	1161–1272	—
Bone (rib)	B0447	I	33428	–10.4	21.0	–4.0	896	28	120	1071–1266	T = 8.2 (5% 3.8)
Textile (hair bundle)	B0447/A03839	I	33508	–11.4	18.6	—	776	31	120	1222–1377	—
Bone (rib)	B0455	I	V-2645-55	–7.9	22.0	–3.6	849	24	18	1189–1274	T = 0.3 (5% 3.8)
Textile (loincloth)	B0455*	I	32883	–17	11.4	—	831	25	18	1211–1280	—

Table 2 Carbon and nitrogen stable isotope values and ^{14}C years for human single samples.

Sample	Inventory	Sector	Lab code OxA-	$\delta^{13}C_{coll}$	$\delta^{15}N$	$\delta^{13}C_{ap}$	^{14}C yr	±	cal AD
Bone (rib)	B0419	G	V-2648-35	–9.9	13.4	–4.5	1041	25	991–1145
Bone (rib)	B0431	G	X-2645-54	–13.9	12.8	–8.2	1192	25	788–987
Bone (rib)	B0483	G	V-2645-56	–13.3	20.3	–8.5	1293	25	682–876
Bone (rib)	B0424	I	V-2645-52	–14.1	24.4	–10.5	1199	25	778–981
Bone (rib)	B0426	I	V-2645-53	–9.4	18.4	–4.4	819	24	1219–1280

Figure 3 Pica 8 textiles: (a) tunic (A03827) associated with individual B0414; (b) hairball found inside mummy's mouth (A03841), associated with individual B0451; (c) bag (*costal*) associated with individual B0492.

The samples were combusted in a CF-IRMS system – Carlo ERBA NA 2000 coupled to a gas source IRMS, Sercon 20/20 (Brock et al. 2010). CO_2 gas was collected and graphitized, and analyzed in the HVEE AMS system at ORAU. The resulting ^{14}C determinations were calibrated using the SHCal atmospheric curve (Hogg et al. 2013) in OxCal v 4.2.4 (Bronk Ramsey 2013).

Statistical analyses were undertaken in The R Project for Statistical Computing (https://www.r-project.org) and SPSS v23.

RESULTS

The results are presented in Table 1 (paired samples) and Table 2 (unpaired samples). As expected, most dates fall inside the temporal range for the LIP (AD 900–1450) spanning from cal AD 993–1414. Unexpected, however, are the very limited offsets in ^{14}C yr observed in four of the paired dates for associated textile and bone samples (Figure 4). This includes the strongly ^{15}N-enriched individuals (with the highest reaching δ^{15}N values of 21–22‰), which were expected to exhibit offsets on the order of some centuries with diets dominated by marine protein. In one pairing (individual B0451) the date for the textile material—a ball of camelid hair placed inside the mouth—is actually significantly older than the bone, by about 270 ^{14}C yr. This may be a curated object, or one encountered accidentally from an earlier context, and retained as an object of some ritual significance. In any case, it is excluded from further consideration.

Leaving aside the above outlier, the largest offset observed between the paired human bone and textile dates is 127 ^{14}C yr. Three other pairings exhibit offsets of 109–120 yr, giving an average offset of 117 ± 9 ^{14}C yr for this group of four. Surprisingly, the δ^{15}N values for these individuals range widely, from 10.9‰ to 21.0‰, spanning nearly the entire range of those sampled for paired dating, as well as incorporating a substantial part of the variation in δ^{15}N seen in the previous study (Santana-Sagredo et al. 2015a). The remaining four paired dates show no offsets, i.e., they can be successfully combined using the R_combine function in OxCal (see Ward and Wilson 1978). This group includes the individual (B0455) with the highest δ^{15}N value of 22.0‰. Single and multiple linear regression analyses show no statistically significant correlations between any of the human stable isotope measurements (δ^{13}C on collagen and apatite, and δ^{15}N on collagen) and the observed offsets in ^{14}C yr (Figure 5). Surprisingly, however, significant positive correlations were observed between the textile δ^{13}C values ($R^2 = 0.606$, $p < 0.05$) and δ^{15}N ($R^2 = 0.572$, $p < 0.05$) values and the associated ^{14}C offsets (Figure 6).

Four of the five results for the remaining unpaired dates on human bone are largely consistent with the Late Intermediate Period (Figure 7). However, one individual (B0483) can be placed at the end of the Formative Period for the Tarapacá region. The results for two other individuals (B0424 and B0431) exhibit probability distributions crossing the boundary between the Late Formative Period and the LIP.

DISCUSSION

Paired ^{14}C Dates and the MRE

The results indicate the presence of two groups, one with a relatively small but significant offset of 117 ± 9 ^{14}C yr, and the other showing no offset at all (excluding the outlier, B0451). This is a surprising outcome, given the previously assumed importance of marine foods in the diets of some individuals at Pica 8, combined with the strong upwelling regime characterizing northern Chile's coast. The estimated ΔR for the late Holocene along this coast is 355 ± 105 yr (Ortlieb et al. 2011; Latorre et al. 2015). When added to the global ocean MRE value of 400 yr, the combined offset should be on the order of 650–860 yr for an individual obtaining most or all of their protein from the sea [see Fernandes et al. (2014) on the use of ^{14}C offsets as a dietary proxy]. Using this range, the observed average offset of 117 ± 9 yr for four individuals implies a contribution of marine foods on the order of between 12.6% and 19.4%, i.e., the observed offset

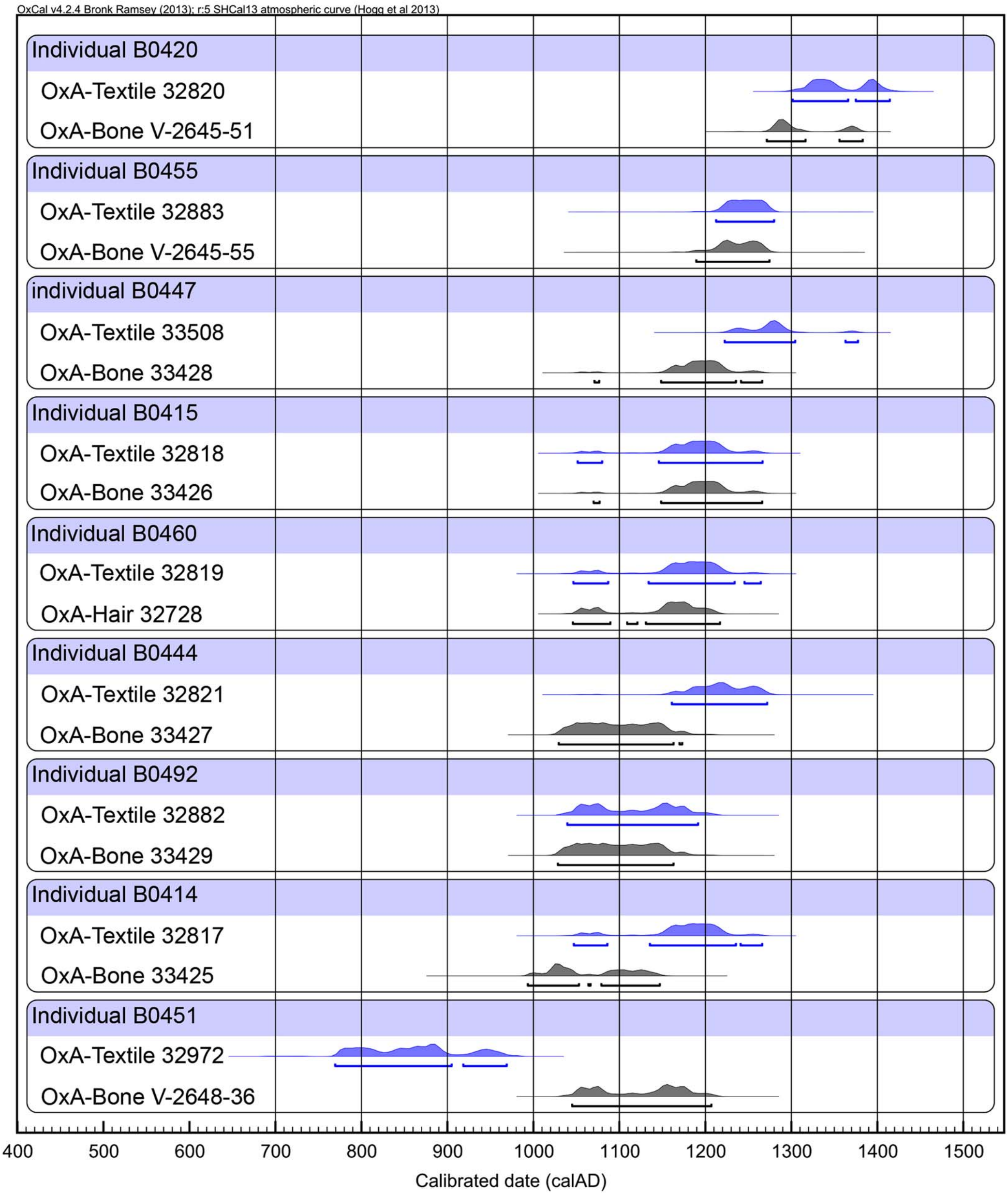

Figure 4 Calibrated paired radiocarbon dates of human bone collagen/hair keratin and camelids textiles/fibers from Pica 8.

in the four paired dates as a proportion of the expected total marine reservoir offset in a 100% marine consumer, taking into account the errors associated with these estimates (Table 3).

$$\text{Minimum } \%\,\text{marine} = {}^{14}\text{C}_{\text{minoffset}}/\text{MRO}_{\text{max}}$$
$$\text{Maximum } \%\,\text{marine} = {}^{14}\text{C}_{\text{maxoffset}}/\text{MRO}_{\text{min}} \quad (1)$$

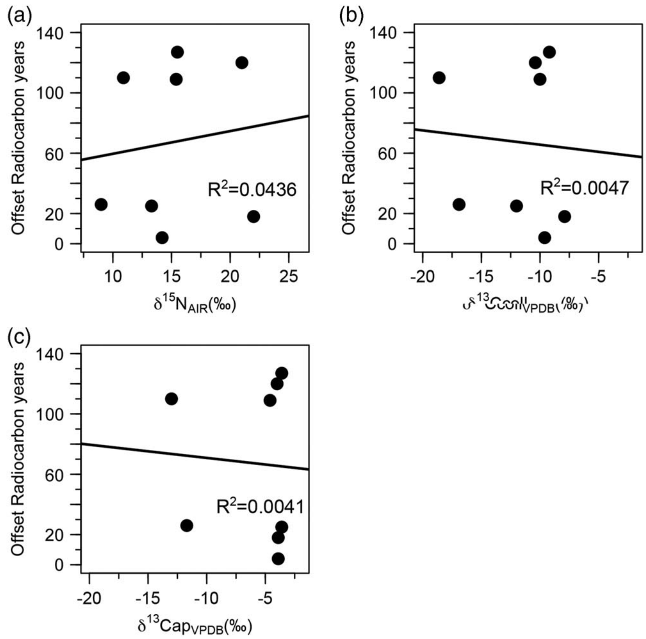

Figure 5 Bivariate plots for (a) $\delta^{15}N$ values, (b) $\delta^{13}C_{coll}$ values, and (c) $\delta^{13}C_{ap}$ values and the offset in ^{14}C yr calculated from the paired bone collagen and textile samples of Pica 8.

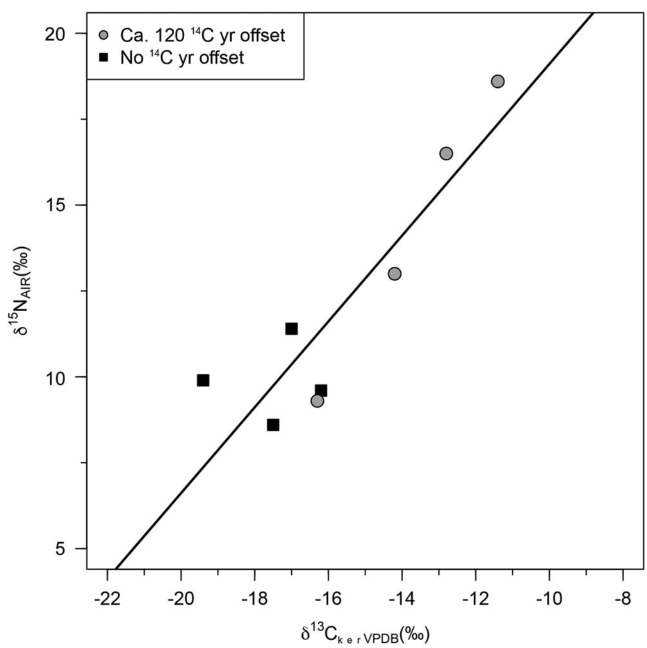

Figure 6 Bivariate plot showing $\delta^{13}C$ and $\delta^{15}N$ values for keratin from textiles and fibers from Pica 8, showing the positive relationship between ^{14}C offset and stable isotope values.

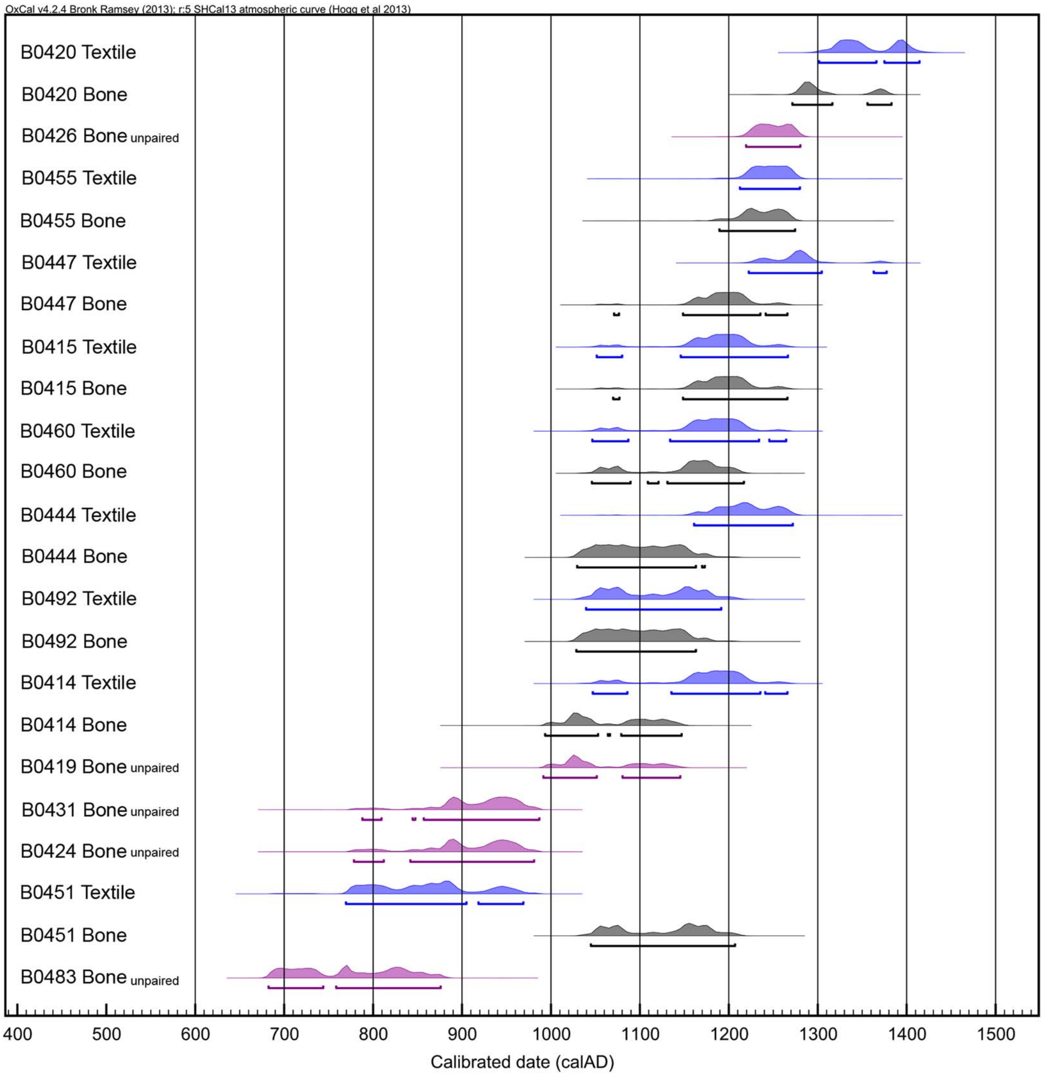

Figure 7 Calibrated dates for paired bone collagen/textile samples and unpaired bone collagen samples from Pica 8.

This result is substantially lower than what would have been predicted on the basis of the stable carbon and nitrogen isotope values (Figure 2). Moreover, the lack of any significant correlation between the stable isotope measurements and the observed ^{14}C offsets is difficult to explain (see below). Certainly, earlier studies have found significant predictive relationships in other contexts (Price et al. 2007; Schulting et al. 2014).

These results challenge the previous interpretation of the high $\delta^{15}N$ values (up to 24.4‰) at Pica 8 as indicating a substantial contribution of marine protein in the diets of these individuals. This was a reasonable assumption, considering the average $\delta^{15}N$ values of between 19.2 ± 2.1‰ and 18.5 ± 1.7‰ for modern northern Chilean marine fish and sea mammals, respectively (Tieszen and Chapman 1992). In addition, there is the aforementioned archaeozoological evidence for marine resources in the cemetery at Pica 8 (Núñez 1984). Other factors, however, could affect

Table 3 Estimation of marine contribution to Pica 8 human diet based on the expected ΔR values for the Late Holocene (Ortlieb et al. 2011) and the average offset in ^{14}C yr obtained for the paired bone-textile dates.

Late Holocene ΔR (355 ± 105)	Total MRO	Observed ^{14}C offset (117 ± 9)	% marine in diet
Max R: 460 yr	860 yr	Min: 108	12.6%
Min R: 250 yr	650 yr	Max: 126	19.4%

the nitrogen isotopic composition of the humans. First, it is well known that dry desert conditions can significantly affect the nitrogen isotopic composition of soils, plants and animals (Evans and Ehleringer 1994; López et al. 2013; Díaz et al. 2016). It has been observed that modern and archaeological Atacama Desert plants can reach δ^{15}N values up to 12‰ (Evans and Ehleringer 1994; Díaz et al. 2016). A similar situation has been observed for archaeological camelids at Tulan, south of the Atacama Salt Lake, also exhibiting relatively high δ^{15}N values above 10‰ (López et al. 2013). Thus, human values of approximately 15‰ could be explained by the consumption of plant or animal resources with high δ^{15}N values, and not necessarily a marine diet. However, there remains a substantial gap between this and the highest observed values of >20‰ that cannot be accounted for solely by aridity effects, so that another explanation is required.

Agriculture was fully developed and consolidated in the south-central Andes by the LIP. Early European chronicles document the use of fertilizers for agricultural activities in the Atacama Desert oases after the Conquest [Frezier 1717; Cieza de León 1922 (1553); Diez de San Miguel 1964 (1567)]. Fertilizers included llama dung, seabird *guano* and anchovy heads (Parsons and Psutty 1975; Julien 1985). Thus, it might be expected that the Pica 8 community fertilized their crops, particularly given the otherwise impoverished soils of the Atacama. The use of any of these manures would have undoubtedly elevated soil ^{15}N composition, in turn reflected in crops and hence in the humans and animals consuming them. In particular, the impact of the use of seabird guano on crops has been clearly demonstrated by the experimental studies carried out by Szpak et al. (2012a, 2012b; 2014). Thus, the consumption of crops fertilized with seabird guano and/or fish would increase human δ^{15}N values significantly. Since the crops would only take up nitrogen from the manure, seabird guano would leave plant ^{13}C and ^{14}C unaffected, effectively uncoupling the two isotopic systems.

Thus, the consumption of crops fertilized with seabird guano presents a viable alternative to marine foods in the explanation of high δ^{15}N values above 20‰. The interpretation of elevated ^{13}C in contexts with access to both C_4 plants (maize) and marine foods is always problematic, and so taking this isotope into consideration does not appear to offer any solutions, except in the case of individuals who clearly partook of neither food. This problem, together with the enormous variability in ^{15}N introduced by the combination of marine foods and manuring with seabird guano, could explain the lack of any predictive relationships between stable isotope measurements and ^{14}C offsets.

The positive correlation (R^2 >0.5) observed for the textile isotope measurements and the offsets in radiocarbon years could be explained by the impact of fertilized crop consumption by these camelids. As mentioned above, the archaeological evidence from the sectors at Pica 8 suggested that some individuals/social units had access to and/or connections with the coast, which could have included the importation of seabird guano for crops. It follows that the camelids owned by these groups would have access to the fertilized, ^{15}N-enriched crops, specifically maize, thus leading to the relatively high δ^{15}N and δ^{13}C values observed for some of the textiles. Humans would not show this correlation since their marine δ^{13}C and δ^{15}N values would overlap with those obtained through consuming the fertilized crops, along with some marine foods that were sufficient to impart a moderate offset in ^{14}C years.

The results obtained here contrast with a case reported by Cases et al. (2008) for northern Chile, in which a considerable offset was observed between an ancient human body and its textile. This individual was found isolated near the María Elena Nitrate Mine in the Atacama Desert. ^{14}C dating of the main textile and a repair patch gave very similar dates (1890 ± 40 BP and 1870 ± 40 BP, respectively). However, the muscle tissue from the individual yielded a ^{14}C determination of 2390 ± 70 BP, 640 yr older than the textiles. This individual also had high $\delta^{13}C_{coll}$ and δ^{15}N values, –13.7‰ and 21.6‰ respectively, comparable to the highest values at Pica 8. In this case the marine diet suggested by the individual's δ^{13}C and δ^{15}N composition, and by the direct association with dry fish and fishhooks (Cases et al. 2008), is confirmed by the observed large ^{14}C offset.

The contrasts observed between this particular case and the Pica 8 paired dates could be explained by temporal and dietary differences. Since this individual dates to the Formative Period, a significant dietary contribution of maize is unlikely as agriculture was still experimental and crops did not form a dietary staple at this time (Santana et al. 2012; García et al. 2014; Santana-Sagredo et al. 2015b). Therefore, the use of seabird guano as fertilizer would not yet have featured, so that the high δ^{15}N value seen in the María Elena individual was likely to be exclusively the result of a marine diet. It is also important to consider that ΔR values can change dramatically through time (Ascough et al. 2004, 2010; Dewar et al. 2012; Hutchinson et al. 2004; Ortlieb et al. 2011; Latorre et al. 2015). For instance, the range of ΔR values reported for northern Chile during the Holocene goes from 48 ± 36 yr to 1052 ±47 yr (Ortlieb et al. 2011). Thus, it is possible that ΔR values in the Formative Period were considerably higher compared to the LIP, which could also have an impact on the differences observed.

Diet Diveristy in Pica 8: Diachronic or Synchronic?

The paired dates obtained here strongly suggest that the isotopic variability observed at Pica 8 is not related to diachronic change. On the contrary, the limited range of dates for all but one individual, of between AD 990 and 1270 yr, suggests that considerable heterogeneity existed within a broadly contemporaneous community. This is further emphasized by the absence of any chronological trends between the stable isotope results and the calibrated dates. However, the lack of a predictive relationship between the stable isotope values and the small marine reservoir offset that was identified for a subset of individuals requires a re-consideration of the source of the isotopic variability at Pica 8. Rather than marine foods as previously proposed (Santana-Sagredo et al. 2015a), it may relate to differential access to seabird guano for manuring of crops, particularly maize, which by the LIP had become an important staple (Santana-Sagredo et al. 2015b). Thus, a link with the coast is still indicated, and no doubt included the transportation of some marine foods, which after all were present in the cemetery and also indicated in the diets of some individuals by the modest ^{14}C offsets. But more importantly for explaining the isotopic variability—most notably the highly enriched ^{15}N—is the access to seabird guano that these exchange links may have facilitated. What is most interesting, then, is that the resulting fertilized crops appear not to have been made available to the entire community at Pica 8. This reinforces the impression obtained from the differing isotopic results in the sectors, that sections of the community maintained distinct and separate diets and life-ways (perhaps kin-based?).

While significant temporal *trends* can be effectively ruled out by the results of the dating program, it is still possible that there were small-scale fluctuations in access to coastal resources (including seabird guano) over a period of some decades—too brief a duration to be detected by ^{14}C. In this scenario, when guano was available it was used, significantly raising the δ^{15}N levels for that generation. But it is hard to see how such fluctuations in access should come about, as the 90-km distance to the coast could hardly be construed as a barrier, particularly given the presence of pack animals and of long-established coast-inland trade connections. So, while acknowledging this possibility, we do not consider it likely.

The textiles at Pica 8 also hint at different origins and connections. For instance, some individuals (B0414, B0415, B0444, and B0460) were interred with textile styles typically associated with the local Pica-Tarapacá Complex (Agüero 2015). However, in this case they cross-cut the different cemetery sectors (B, D, and F). The hair bundle associated with individual B0447 is a common trait found on the Tarapacá coast. This individual was buried in sector I where most of the marine evidence was found, providing a δ^{15}N value of 21‰, and an offset of 120 ^{14}C yr in the paired dating. Yet, since other individuals with equally high δ^{15}N values show no offset, this in itself cannot be interpreted as indicating access to marine foods.

One male individual (B0492) from sector D was associated with a "non-local" bag of the type used for camelids to carry and store objects and food (known as *costal*) (Cases et al. 2007). The textile style has also been observed by one of the authors (CA) in the San Pedro Oases, specifically in Quitor and Solcor (Agüero 2004). However, it is not possible to associate it directly with the San Pedro de Atacama groups, since it could also have an origin in the highlands or elsewhere. Strikingly, this individual shows carbon and oxygen isotopic values that are clearly non-local, and are not inconsistent with highland origins, marking him as a clear outlier in Pica 8 (Santana-Sagredo et al. 2015a).

The most peculiar case is individual B0451 with the hairball found inside the mouth. This was an ancestral practice dating to the Formative Period in the Tarapacá coast and indeed the date date for the hairball (AD 796–969) is consistent with the Formative Period. One possible explanation is that it was removed from a burial in the coast and brought inland to the Pica oases. An alternative would be long-term curation of the hairball and re-use during the LIP. In any case, in Tarapacá it is associated with a typical coastal practice.

Individual B0420 has a relatively low δ^{15}N value of 10.9‰ and is also an outlier for carbon and oxygen isotopes (Santana-Sagredo et al. 2015a). At cal AD 1271–1383, this is the most "recent" of the dated individuals. However, the camelid textile associated with this individual has an offset of 110 yr (cal AD 1301–1414) compared to the human bone collagen, suggesting the consumption of approximately 15% marine protein by the human, but then this seems incommensurate with the δ^{15}N value. Unfortunately, this textile (possibly a bag) in particular was in a poor state of preservation and it not possible to identify its type, style, and cultural tradition.

The only dates that could suggest temporal differences in the cemetery are individuals B0483, B0424, and B0431 for which we have no paired textile dates. Individual B0483 (cal AD 682–876) clearly falls in the Late Formative Period for the Tarapacá region, while individuals B0424 (cal AD 778–981) and B0431 (cal AD 788–987) present dates with probability distributions straddling the Late Formative Period to LIP boundary. It is possible that both are affected to a small extent by the MRE. A high marine diet was previously suggested for individual B0424 given the high $\delta^{13}C_{coll}$ and $\delta^{15}N$ values of –14.1‰ and 24.4‰, respectively, together with a $\delta^{13}C_{apatite}$ value of –10.5‰ sufficiently low to suggest minimal maize in the diet (Santana-Sagredo et al. 2015a). However, given the results of the paired dating program, the inference of a high marine diet is now potentially problematic. Nevertheless, the fact that half of the individuals in the paired dating exercise exhibited offsets of approximately 100 ^{14}C yr makes a moderate contribution of marine foods a relatively uncontroversial claim.

As can be seen, the cultural variability of Pica 8 is reflected in the dietary composition of the population together with the textile styles associated with the burials. The dates clearly demonstrate that the isotopic and dietary heterogeneity is not diachronic in origin, supporting instead the presence of a multicultural community at Pica 8 during the LIP, maintaining distinct practices over some centuries at least. Yet these individuals, who consumed different diets and were interred with diverse textile types and styles, were buried in the same cemetery. Thus, identity and ethnicity factors appear to have played a major role in Pica during the LIP, leading to the coexistence (and presumably cohabitation) of different cultural groups.

CONCLUSIONS

Paired human–textile ^{14}C dating from 9 graves at Pica 8 were undertaken as a first step to understanding the chronology of the Pica-Tarapacá Cultural Complex and the potential impact of the MRE on the dating of human remains from this region and period. The results show two clear groups: one with an approximately 100 ^{14}C-yr offset and the other with no offset. The former is consistent with a contribution of approximately 12–19% of marine protein, not insignificant but considerably less than was expected. Moreover, no correlations were observed between the high $\delta^{15}N$ values—or indeed with $\delta^{13}C$ measurements on collagen and apatite—and the ^{14}C offsets between the paired dates. These results question the use of stable nitrogen isotopes in order to evaluate the MRE on the ^{14}C dates for the LIP in northern Chile, and potentially more widely (e.g. southern Peru) and for other periods (e.g. Inca). Use of marine fertilizers and the possible effects of aridity could strongly influence the nitrogen isotopic composition of crops, animals and ultimately humans. Since fertilizers would not significantly affect the carbon composition of the crops, no impact of the MRE would be observed on them nor on the individuals consuming them. This, then, is a possible explanation for those individuals with high $\delta^{15}N$ values and no ^{14}C offset. The MRE for northern Chile and southern Peru remains poorly known and this situation greatly complicates the dating and interpretation of samples associated with the coast. We predict that the effect found in this paper will be largely restricted to the LIP and later periods when maize agriculture had become important and intensive, but of limited importance in the preceding Formative period.

The main question posed in the paper was whether the previously noted high isotopic/dietary diversity was related to temporal change, or whether it reflected variation within a living community. The results appear to favor the latter explanation, with no relationship being found between the stable isotope measurements and radiocarbon determinations. This in turn suggests the importance of sociocultural influences such as identity, ethnicity, and inequality in structuring foodways, and in determining differential access to marine resources, both in the form of food but also in the availability of seabird guano to fertilize the crops that were intensively cultivated at the Pica oasis. The alternative explanation that access to seabird guano was intermittent on short timescales not amenable to ^{14}C dating and therefore undetectable seems improbable, since the advantages of the system once established should have been readily apparent. It does, however, raise the question of why not everyone in the community had access to fertilized crops.

This situation requires us to rethink social complexity in the different Andean territories before the Inca expansion. Thus, while answering one question, we have raised others—not an unusual outcome in archaeology or indeed any science.

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