**Reassessing the late prehistory of the semiarid north of Chile: Diet, mobility, and chronology of individuals buried at the El Olivar**

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**Appendix 2**

**Sampling**

Dental samples (10 mg of enamel of second or third molars) from 58 individuals, and bone samples (1-3 g from ribs or long bones) from 60 individuals were sent to the Cornell University Stable Isotope Laboratory (COIL). Before extractions of dental samples, each tooth was photographed and 3D models were generated for virtual visualization and from which it is possible be printed. Bone samples were photographed, and registered in the charts of bioanthropological analysis of each individual.

Bone samples (1-3 g from ribs or long bones) from 20 camelids and 42 humans were sent to the Oxford Radiocarbon Accelerator Unit (ORAU). Bone samples were photographed, and registered in the charts of zooarchaeological and bioanthropological analysis of each specimen/individual.

**Methods at Cornell University Stable Isotope Laboratory (COIL)**

*Collagen Extraction Method for bone samples*

a) Grinding into powder using a mortar and pestle.

b) Combined 0.5 g powdered bone and 5 ml 0.5 Ma HCl in a 50 ml centrifuge tube. React for 30 min. at room temperature.

c) Centrifuge (5 min/2500 rpm) and thoroughly rinse with H2O (deionized water roughly 5.6) to neutrality (wash x2). Centrifuging again after each rinse.

d) Discard supernatant, and react 5 ml 0.1 Mb NaOH with the remaining pellet for 30 min.

e) Centrifuge and rinse to neutrality with H2O (wash x3).

f) Discard supernatant and fill centrifuge tube with pH 3.0c water to ~10ml and react 24 hours at ~80oC with the lid closed.

g) Centrifuge and keep supernatant.

h) Freeze-dry the remaining solution.

Necessary calculations:

a To make 250ml stock of 0.5 M HCl: combine 10.3ml 12.1 M HCl with 239.7 ml H2O.

+Calculation: 250 ml of 0.5 M HCl should contain a total of 0.125 moles of HCl (0.5mol/L\*0.25L=0.125 moles)

+The concentrated acid contains 12.1moles/L

+To calculate the volume of concentrated acid to use, multiply the desired number of total moles in the 0.5M solution by the inverse of the molarity in the concentrated solution: 1L/12.1moles \* 0.125moles\*1000ml/L=10.3ml of concentrated acid to be used.

b To make 250 ml stock of 0.1 M NaOH from NaOH pellets: combine 1g NaOH with 250 mL H2O.

+NaOH = 40 g/mol

+40g/mol \* 0.1mol/L = 4g/L; 4g/L \* 0.25L=1g total

c To make 2.5 L stock of pH=3.0 H2O, using concentrated HCl: this is a hydrogen concentration of 10^-3 moles/L. HCl dissociates completely, so moles HCl added to solution = moles H+ added to solution. Acidity from CO2 can be ignored for practical purposes (pH = 2.9989 vs. 3.0). Slight variation in final pH is OK (i.e. 2.5-3.5).

+0.001moles/L \* 2.5L = 0.0025moles acid

+1L/12.1moles \* 0.0025 moles \* 1000000ul/L = 206.6 ul HCl into the DI wáter or add HCl until pH is 3.0, stir well between measurements

Method adapted from:

Ambrose SH (1990) Preparation and characterization of bone and tooth collagen for stable carbon and nitrogen isotope analysis.  *Journal of Archaeological Science* **17**, 430-451

Lambert JB, Grupe G (1993) *Prehistoric Human Bone – Archaeology at the Molecular Level.* Springer-Verlag, 1-130.

The collagen fragments were processed and ground, and then demineralized in a hydrochloric acid solution. Collagen extraction was completed by lyophilizing each sample. The δ13Ccol and δ15Ncol analyses were performed on a Thermo Delta V Isotope Ratio Mass Spectrometer interfaced to an NC2500 Elemental analyser. These results were calibrated in relation to internal standards (“Deer” and “Methionine”). In the case of analyses based on apatite, the bone subsamples and those of tooth enamel were pulverized and treated in an acetic acid solution, to remove the organic fraction of the bone and possible contamination by secondary carbonates of teeth. Subsequently, samples of δ13Cap and δ18Oap were analysed using a Thermo Delta V Isotope Ratio Mass Spectrometer, with an interface to Gas Bench II. The results were calibrated in relation to the international standards of the IAEA (International Atomic Energy Agency), NBS-18 and NBS-19.

**Methods at Oxford Radiocarbon Accelerator Unit (ORAU), University of Oxford**

Bone collagen extraction was carried out following the ultrafiltration method. Pretreatment, target preparation measured using *accelerator* mass spectrometry (AMS), were carried out following procedures detailed in Bronk Ramsey et al. (2004) and Brock et al. (2010).

ORAU has four stable isotope mass spectrometers. Two Europa Scientific instruments are used in conjunction with automated gas collection systems for radiocarbon samples. Another two (one from Finingan and one from Europa Scientific) that are used for stable isotope research. All are usually operated in continuous flow mode (where the sample is transported in a stream of inert He gas). They are set up to measure the carbon and nitrogen compositions and stable isotope ratios. Precisions are usually better 0.1 to 0.2 per mil for δ13C and 0.3 per mil for δ15N (<https://c14.arch.ox.ac.uk/methods.html#chem>).

References

Bronk Ramsey B, Higham T, Leach P (2004). Towards high-precision AMS: Progress and limitations. *Radiocarbon* **46** (1), 17-24.

Brock F, Higham T, Ditchﬁeld P, Bronk Ramsey C (2010). Current pretreatment methods for AMS radiocarbon dating at the Oxford Radiocarbon Accelerator Unit (ORAU). *Radiocarbon* **52** (1): 103-12.

**Comparison of human δ18O values with values of waters sources of the area**

The human δ18OVPDB values were converted into drinking water values (δ¹⁸Odw) utilising the conversion equations indicated by Coplen (1988), Daux et al. (2008), and Chenery et al. (2012) (Table 1). The δ¹⁸OSMOW values of waters sources of the area can be grouped into three main sectors from the west (coast) to the east (Cordillera de los Andes) (Squeo et al. 2006; Strauch et al. 2006) (Table 2).

Table 1. δ¹⁸O human values converted into drinking water values.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Individual** | **δ18OVPDB** | **δ18Odw** |  | **Individual** | **δ18OVPDB** | **δ18Odw** |
| 4 | -10.99 | -17.5 |  | 127 | -9.43 | -15.0 |
| 9 | -10.70 | -17.0 |  | 135 | -10.16 | -16.1 |
| 20 | -10.28 | -16.3 |  | 149 | -8.87 | -14.0 |
| 24 | -9.36 | -14.8 |  | 156 | -8.13 | -12.8 |
| 25 | -10.08 | -16.0 |  | 161 | -9.89 | -15.7 |
| 27 | -9.31 | -14.8 |  | 163 | -9.17 | -14.5 |
| 34 | -9.90 | -15.7 |  | 164 | -9.70 | -15.4 |
| 41 | -10.79 | -17.2 |  | 165 | -7.98 | -12.6 |
| 57 | -9.33 | -14.8 |  | 166 | -9.94 | -15.8 |
| 63 | -10.66 | -17.0 |  | 169 | -9.40 | -14.9 |
| 69 | -9.48 | -15.0 |  | 173 | -8.91 | -14.1 |
| 74 | -8.63 | -13.6 |  | 181 | -9.02 | -14.3 |
| 76 | -7.28 | -11.4 |  | 183 | -9.05 | -14.3 |
| 77 | -8.93 | -14.1 |  | 191 | -9.26 | -14.7 |
| 82 | -10.89 | -17.3 |  | 194 | -9.29 | -14.7 |
| 97 | -9.37 | -14.8 |  | 196 | -9.34 | -14.8 |
| 99 | -9.16 | -14.5 |  | 197 | -8.72 | -13.8 |
| 105 | -9.32 | -14.8 |  | 208 | -9.95 | -15.8 |
| 113 | -8.36 | -13.2 |  | 214 | -9.80 | -15.5 |
| 122 | -7.61 | -12.0 |  |  |  |  |

Table 2. δ¹⁸OSMOW values of water from the area of study. Masl, meters above sea level

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
|  | Coast | | Valley | | Cordillera de los Andes |
| Elqui Area  (29° 43´ to  30° 20´S) | -1‰ to -3‰ morning fog | -4.8‰ rain | >-14‰ (river)  below 500 masl | -13‰ to -15 ‰ (river)  500-1500 masl | -16‰ to -17‰ (river)  above1500 masl |
| groundwater  -4‰ to -8‰ from low basin  -10.5‰ to -12‰ from high basin | | groundwater with similar values | | |

References

Chenery CA, Pashley V, Lamb AL, Sloane HJ, Evans JA (2012) The oxygen isotope relationship between the phosphate and structural carbonate fractions of human bioapatite. *Rapid Communications in Mass Spectrometry* **26,** 309-319.

Coplen T (1988) Normalization of oxygen and hydrogen isotope data. *Chemical Geology: Isotope Geoscience section* **72**, 293-297.

Daux V, Lécuyer C, Héran MA, Amiot R, SimonL, Fourel F, Martineau F, Lynnerup N, Reychler H, Escarguel G (2008). Oxygen isotope fractionation between human phosphate and water revisited. *Journal of Human Evolution* **55**, 1138-1147.

Squeo FA, Aravena R, Aguirre E, Pollastri A, Jorquera C, Ehleringer JR (2006) Groundwater dynamics in a coastal aquifer in north-central Chile: Implications for groundwater recharge in an arid ecosystem. *Journal of Arid Environments* **67**, 240-254.

Strauch G, Oyarzún J, Fiebig-Wittmaack M, González E, Weise SM (2006) Contributions of the different water sources to the Elqui river runoff (northern Chile) evaluated by H/O isotopes. *Isotopes in Environmental and Health Studies* **42**, 303-322.

**Calculation of marine diet percentages**

The percentages of marine diet were estimate from our database of stable isotopes compositions (δ13C and δ15N) of archaeological resources recovered at the El Olivar (not published, but in ongoing), using MixSIAR Bayesian isotope mixing models (Stock and Semmens 2016; Stock et al. 2018). Human δ13C and δ15N values were adjusted by 2.6 ± 0.35 for carbon and 3.1 ± 0.26 for nitrogen to compensate for tissue-diet discrimination and the trophic level effect, respectively. This taking into account the results of Smith et al. (2017) in archaeological coastal groups of Northern Chile.

References

Smith EK, Pestle WJ, Clarot A, Gallardo F (2017) Modelling breastfeeding and weaning practices (BWP) on the coast of Northern Chile's Atacama Desert during the formative period. *The Journal of Island and Coastal Archaeology* **12**(4), 558-571.

Stock BC, Jackson AL, Ward EJ, Parnell AC, Phillips DL, Semmens BX (2018) Analyzing mixing systems using a new generation of Bayesian tracer mixing models. *PeerJ* **6**, e5096. https://doi.org/10.7717/peerj.5096.

Stock BC, Semmens BX (2016). “MixSIAR GUI User Manual.” doi:10.5281/zenodo.1209993, Version 3.1, <https://github.com/brianstock/MixSIAR>

**OxCal Codes**

The OxCal runfiles employed 1536 AD as constraint (*ante quem*)

**Secondary Burials**

Sequence()

{

Curve("SHCal20","shcal20.14c");

Curve("Marine20","marine20.14c");

Boundary();

Phase()

{

Delta\_R("LocalMarine10",-55,25);

Mix\_Curves("Mixed10","SHCal20","LocalMarine10",49.9,10);

R\_Date("ES 041",810,17);

Curve("=Marine20");

Delta\_R("LocalMarine5",-55,25);

Mix\_Curves("Mixed5","SHCal20","LocalMarine5",48.3,10);

R\_Date("ES 009",766,17);

Curve("=Marine20");

Delta\_R("LocalMarine3",-55,25);

Mix\_Curves("Mixed3","SHCal20","LocalMarine3",52.7,10);

R\_Date("ES 004",745,19);

Curve("=Marine20");

Delta\_R("LocalMarine2",-55,25);

Mix\_Curves("Mixed2","SHCal20","LocalMarine2",60.5,10);

R\_Date("ES 003",782,17);

Curve("=Marine20");

Delta\_R("LocalMarine7",-55,25);

Mix\_Curves("Mixed7","SHCal20","LocalMarine7",53.5,10);

R\_Date("ES 022",742,17);

Curve("=Marine20");

Delta\_R("LocalMarine6",-55,25);

Mix\_Curves("Mixed6","SHCal20","LocalMarine6",71.6,10);

R\_Date("ES 020",834,18);

Curve("=Marine20");

Delta\_R("LocalMarine1",-55,25);

Mix\_Curves("Mixed1","SHCal20","LocalMarine1",54,10);

R\_Date("ES 001",735,18);

Curve("=Marine20");

Delta\_R("LocalMarine8",-55,25);

Mix\_Curves("Mixed8","SHCal20","LocalMarine8",57.7,10);

R\_Date("ES 027",715,17);

Curve("=Marine20");

Delta\_R("LocalMarine9",-55,25);

Mix\_Curves("Mixed9","SHCal20","LocalMarine9",67.9,10);

R\_Date("ES 033",690,17);

Curve("=Marine20");

Delta\_R("LocalMarine4",-55,25);

Mix\_Curves("Mixed4","SHCal20","LocalMarine4",66.7,10);

R\_Date("ES 007",676,17);

};

Date("Almagro", 1536);

};

**Primary Burials**

Sequence()

{

Curve("SHCal20","shcal20.14c");

Curve("Marine20","marine20.14c");

Boundary();

Phase()

{

Delta\_R("LocalMarine1",-55,25);

Mix\_Curves("Mixed1","SHCal20","LocalMarine1",37.6,10);

R\_Date("Ind 003",968,19);

Curve("=Marine20");

Delta\_R("LocalMarine25",-55,25);

Mix\_Curves("Mixed25","SHCal20","LocalMarine25",58.3,10);

R\_Date("Ind 149",948,18);

Curve("=Marine20");

Delta\_R("LocalMarine17",-55,25);

Mix\_Curves("Mixed17","SHCal20","LocalMarine17",23.9,10);

R\_Date("Ind 084",817,17);

Curve("=Marine20");

Delta\_R("LocalMarine34",-55,25);

Mix\_Curves("Mixed34","SHCal20","LocalMarine34",51.9,10);

R\_Date("Ind 194",869,19);

Delta\_R("LocalMarine32",-55,25);

Mix\_Curves("Mixed32","SHCal20","LocalMarine32",49.8,10);

R\_Date("Ind 187",853,17);

Curve("=Marine20");

Delta\_R("LocalMarine28",-55,25);

Mix\_Curves("Mixed28","SHCal20","LocalMarine28",52.8,10);

R\_Date("Ind 165",831,17);

Curve("=Marine20");

Delta\_R("LocalMarine30",-55,25);

Mix\_Curves("Mixed30","SHCal20","LocalMarine30",34.3,10);

R\_Date("Ind 177",684,17);

Curve("=Marine20");

Delta\_R("LocalMarine22",-55,25);

Mix\_Curves("Mixed22","SHCal20","LocalMarine22",47.5,10);

R\_Date("Ind 123",745,17);

Curve("=Marine20");

Delta\_R("LocalMarine19",-55,25);

Mix\_Curves("Mixed19","SHCal20","LocalMarine19",85.4,10);

R\_Date("Ind 094",900,19);

Curve("=Marine20");

Delta\_R("LocalMarine12",-55,25);

Mix\_Curves("Mixed12","SHCal20","LocalMarine12",54.6,10);

R\_Date("Ind 058",758,20);

Curve("=Marine20");

Delta\_R("LocalMarine9",-55,25);

Mix\_Curves("Mixed9","SHCal20","LocalMarine9",77.6,10);

R\_Date("Ind 032",850,19);

Curve("=Marine20");

Delta\_R("LocalMarine31",-55,25);

Mix\_Curves("Mixed31","SHCal20","LocalMarine31",49.2,10);

R\_Date("Ind 183",735,17);

Curve("=Marine20");

Delta\_R("LocalMarine13",-55,25);

Mix\_Curves("Mixed13","SHCal20","LocalMarine13",65.1,10);

R\_Date("Ind 074",797,19);

Curve("=Marine20");

Delta\_R("LocalMarine24",-55,25);

Mix\_Curves("Mixed24","SHCal20","LocalMarine24",50.5,10);

R\_Date("Ind 142",733,17);

Curve("=Marine20");

Delta\_R("LocalMarine16",-55,25);

Mix\_Curves("Mixed16","SHCal20","LocalMarine16",53.4,10);

R\_Date("Ind 082",739,19);

Curve("=Marine20");

Delta\_R("LocalMarine5",-55,25);

Mix\_Curves("Mixed5","SHCal20","LocalMarine5",60.3,10);

R\_Date("Ind 024",766,18);

Curve("=Marine20");

Delta\_R("LocalMarine11",-55,25);

Mix\_Curves("Mixed11","SHCal20","LocalMarine11",57.8,10);

R\_Date("Ind 041",749,19);

Curve("=Marine20");

Delta\_R("LocalMarine20",-55,25);

Mix\_Curves("Mixed20","SHCal20","LocalMarine20",59.1,10);

R\_Date("Ind 099",757,17);

Curve("=Marine20");

Delta\_R("LocalMarine21",-55,25);

Mix\_Curves("Mixed21","SHCal20","LocalMarine21",78.2,10);

R\_Date("Ind 122",831,18);

Curve("=Marine20");

Delta\_R("LocalMarine33",-55,25);

Mix\_Curves("Mixed33","SHCal20","LocalMarine33",61.3,10);

R\_Date("Ind 191",746,17);

Curve("=Marine20");

Delta\_R("LocalMarine8",-55,25);

Mix\_Curves("Mixed8","SHCal20","LocalMarine8",65.9,10);

R\_Date("Ind 029",756,19);

Curve("=Marine20");

Delta\_R("LocalMarine29",-55,25);

Mix\_Curves("Mixed29","SHCal20","LocalMarine29",58.3,10);

R\_Date("Ind 169",723,19);

Curve("=Marine20");

Delta\_R("LocalMarine4",-55,25);

Mix\_Curves("Mixed4","SHCal20","LocalMarine4",64.8,10);

R\_Date("Ind 020",740,19);

Curve("=Marine20");

Delta\_R("LocalMarine15",-55,25);

Mix\_Curves("Mixed15","SHCal20","LocalMarine15",63,10);

R\_Date("Ind 077",729,19);

Curve("=Marine20");

Delta\_R("LocalMarine27",-55,25);

Mix\_Curves("Mixed27","SHCal20","LocalMarine27",56.6,10);

R\_Date("Ind 163",698,17);

Curve("=Marine20");

Delta\_R("LocalMarine26",-55,25);

Mix\_Curves("Mixed26","SHCal20","LocalMarine26",62.5,10);

R\_Date("Ind 156",721,17);

Curve("=Marine20");

Delta\_R("LocalMarine6",-55,25);

Mix\_Curves("Mixed6","SHCal20","LocalMarine6",47.8,10);

R\_Date("Ind 027",634,19);

Curve("=Marine20");

Delta\_R("LocalMarine10",-55,25);

Mix\_Curves("Mixed10","SHCal20","LocalMarine10",66.2,10);

R\_Date("Ind 034",691,18);

Curve("=Marine20");

Delta\_R("LocalMarine23",-55,25);

Mix\_Curves("Mixed23","SHCal20","LocalMarine23",68.8,10);

R\_Date("Ind 138",678,19);

Curve("=Marine20");

Delta\_R("LocalMarine3",-55,25);

Mix\_Curves("Mixed3","SHCal20","LocalMarine3",67.7,10);

R\_Date("Ind 013",638,17);

Curve("=Marine20");

Delta\_R("LocalMarine2",-55,25);

Mix\_Curves("Mixed2","SHCal20","LocalMarine2",69.4,10);

R\_Date("Ind 005",625,19);

Curve("=Marine20");

Delta\_R("LocalMarine18",-55,25);

Mix\_Curves("Mixed18","SHCal20","LocalMarine18",71.4,10);

R\_Date("Ind 092",641,17);

Curve("=Marine20");

};

Date("Almagro", 1536);

};

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