Boiled or roasted? Bivalve cooking methods of early Puerto Ricans elucidated using clumped isotopes

Philip T. Staudigel1,2*, Peter K. Swart3, Ali Pourmand3, Carmen A. Laguer-Díaz3, William J. Pestle4

Cooking technique reflects a combination of cultural and technological factors; here, we attempt to constrain bivalve cooking temperatures for a pre-Columbian Puerto Rican native population using carbonate clumped isotopes. Analyses of 24 bivalve specimens (Phacoides pectinatus) from a shell midden in Cabo Rojo, Puerto Rico, suggest that samples were heated up to 200°C, indicating that roasting rather than boiling may have been the preferred cooking technique. More than half of analyzed samples exhibited a distinct change from modern uncooked shells, possibly reflecting different cooking techniques or the use of a single method wherein shells are unevenly heated, such as when placed on a heated surface. Roasting bivalves would not necessitate the use of ceramic technologies, an observation concurrent with the absence of such artifacts at this site.

INTRODUCTION

Investigation of ancient culinary techniques gives valuable insight into the technologies available to ancient cultures. Food preparation techniques can be studied by examination of written accounts (1, 2), excavated food preparation technology (3–5), and chemical analyses of archaeological sites and food waste (5–8). Here, we present a study wherein peak cooking temperatures of the bivalve Phacoides pectinatus, recovered from a pre-Columbian shell midden in Cabo Rojo, Puerto Rico (Fig. 1), are determined using the distribution of clumped isotope bonds (13C–18O) in the aragonite shells. The enrichment of 13C–18O bonds in CO2 liberated from carbonate is expressed using the parameter Δ47, which is defined as follows

$$\Delta_{47} = \left[ \left( \frac{R_{47}^\text{measured}}{R_{47}^\text{stochastic}} - 1 \right) - \left( \frac{R_{46}^\text{measured}}{R_{46}^\text{stochastic}} - 1 \right) \right] \times 1000 \%$$

RX refers to the ratio of mass-X CO2 relative to mass-44, as measured using isotope ratio mass spectrometry, and the subscript refers to measured values or predicted ratios in a stochastically distributed gas.

The motivation of this study was to determine whether boiling, which may require the use of ceramic vessels, or roasting, which has no substantial technological requirement, was the preferred cooking method for the early inhabitants of Puerto Rico. Recent studies (9, 10) investigating the abundance of clumped isotope bonds (18O–13C) in the CO32− ion in biogenic aragonite (CaCO3) reveal that the moderate heating (125° to 250°C) used in common culinary preparation techniques is sufficient to change the abundance of these bonds. The extent of this alteration is proportional to the duration of heating (125° to 250°C) used in common culinary preparation methods for the early inhabitants of Puerto Rico.

RESULTS

All shell midden constituents were analyzed using X-ray diffractometry (XRD) and confirmed to be >95% aragonite (23); the nominal precision of this method is ±5%. The implementation of a separate
calibration scheme (discussed in the Supplementary Materials) results in a change in calcite content by less than 1% from the aforementioned values. Conversion from aragonite to calcite would imply either post-depositional dissolution of aragonite and subsequent precipitation of calcite or heating in excess of 350°C, which would facilitate the solid-state conversion from one mineral to another accompanied by a significant decrease in the materials’ ∆47 values (10). At 350°C, ~10% of aragonite will convert to calcite over approximately 30 min; at 400°C, this same degree of alteration would occur over approximately 5 min (10). The absence of conversion from aragonite to calcite has historically been inferred to represent that these alteration processes never occurred (11) and thus any heating was below the threshold for the mineral conversion.

The isotopic compositions of all shell samples are presented in Table 1. The carbon isotope ratio (δ13C) of shell midden constituents did not vary significantly between ancient samples, although modern shells were more variable in composition, a result likely attributable to changes in land utilization in the region, which could affect relative supply of marine carbon and organic carbon. The oxygen isotope ratios (δ18O) were not statistically significantly different given the number of analyses (P > 0.05). The average δ18O value in all samples was ~0.1‰ relative to the Vienna Pee Dee belemnite (PDB) standard and ranged between 0.7 and ~0.7‰, respectively.

Modern samples’ ∆47 values ranged between 0.70 and 0.67‰ in the thermodynamically defined absolute reference frame (24). On the same scale, bivalves collected from the shell midden ranged between 0.71 and 0.51‰, with 10 falling within the range of modern samples and 14 exceeding this range toward more negative values (Fig. 3A). ∆47 values for shells in Stratum I were significantly more variable than modern specimens, and F values (σmidden2/σmodern2) for shell midden samples compared to modern samples range between 15 and 44 (n = 6 for each population, P = 0.005 to 0.0003). Levels 1.1 through 1.4 all had statistically similar variance to one another. A combined t test between all samples recovered from Stratum I (n = 24) and the control group shells (n = 6) indicates a highly significant (P < 0.001)
difference in mean $\Delta_{47}$ value between the two populations. There is no significant difference between the left and right valves' $\Delta_{47}$ values among shell midden constituents.

**DISCUSSION**

The $\Delta_{47}$ values of the modern control group bivalves fall within a narrow range between +0.70 and +0.67‰, which corresponds to a temperature range of 23° to 32°C when using the temperature-$\Delta_{47}$ calibration generated using the same instrumentation (25). This temperature range is consistent with seasonal variation in the region (26). In contrast, the $\Delta_{47}$ values in the shells from the shell midden corresponded to paleotemperatures between 21° and 118°C. Evidently, the warmer temperature estimates significantly exceed the natural temperature range, indicating that the $\Delta_{47}$ values may have been reequilibrated during a heating period, which we interpret as having occurred during cooking before consumption and deposition. Whereas wildfires are a distinct possibility at this site, the compact and relatively unmixed stratigraphy of the deposit and lack of highly thermally altered shells in the study sample argues against the occurrence of high-temperature (up to 800°C) grass fires; thus, post-burial heating was likely minimal and unlikely to have affected the $\Delta_{47}$ values of shell midden constituents. Of the 24 shells analyzed, 14 have $\Delta_{47}$ values less than the average ± 1 SD for modern-day shells.

### Table 1. Average $\delta^{13}$C, $\delta^{18}$O, and $\Delta_{47}$ values for all 30 sample materials. Uncertainty corresponds to ±1 SD from replicate analyses. Inferred paleotemperatures were calculated from $\Delta_{47}$ values using the calibration from Staudigel et al. (25). ARF, absolute reference frame; VPDB, Vienna PDB.

| Sample group | ID | N | $\delta^{13}$C (%VPDB) ± (‰VPDB) | $\delta^{18}$O (%VPDB) ± (‰VPDB) | $\Delta_{47}$ (%ARF) ± (°C) | Inferred paleotemperature (°C) |
|--------------|----|---|-------------------------------|-------------------------------|----------------------------|-------------------------------|
| **CR_Modern** |    |   |                               |                               |                            |                               |
| A            | 2  | 3.558 | 0.052 | -0.126 | 0.092 | 0.673 | 0.004 | 32.7 |
| B            | 1  | -2.775 | 0.166 | 0.692 | 26.1 |
| C            | 1  | -2.911 | 0.280 | 0.675 | 32.0 |
| D            | 2  | 3.196 | 0.025 | 0.307 | 0.130 | 0.687 | 0.000 | 27.8 |
| E            | 1  | 4.708 | -0.448 | 0.701 | 23.2 |
| F            | 1  | 1.730 | 0.710 | 0.694 | 25.4 |
| **CRNWR_P13 (shell midden) samples** |    |   |                               |                               |                            |                               |
| Level 1.1 A  | 1  | 3.413 | -0.097 | 0.687 | 27.8 |
| Level 1.1 B  | 1  | 2.954 | -0.431 | 0.687 | 27.8 |
| Level 1.1 C  | 1  | 2.866 | 0.195 | 0.681 | 29.8 |
| Level 1.1 D  | 2  | 4.244 | 0.033 | -0.570 | 0.012 | 0.598 | 0.004 | 63.3 |
| Level 1.1 E  | 2  | 4.412 | 0.021 | -0.429 | 0.044 | 0.597 | 0.008 | 64.1 |
| Level 1.1 F  | 2  | 4.241 | 0.069 | -0.077 | 0.031 | 0.639 | 0.010 | 45.3 |
| Level 1.2 A  | 2  | 3.123 | 0.079 | 0.674 | 0.019 | 22.1 |
| Level 1.2 B  | 1  | 3.679 | 0.061 | 0.660 | 37.2 |
| Level 1.2 C  | 2  | 3.776 | 0.005 | -0.748 | 0.049 | 0.507 | 0.044 | 117.6 |
| Level 1.2 D  | 1  | 4.029 | -0.395 | 0.679 | 30.5 |
| Level 1.2 E  | 2  | 3.494 | 0.007 | 0.084 | 0.083 | 0.639 | 0.001 | 45.6 |
| Level 1.2 F  | 2  | 3.234 | 0.152 | 0.113 | 0.075 | 0.573 | 0.027 | 76.0 |
| Level 1.3 A  | 2  | 2.378 | 0.091 | 0.168 | 0.143 | 0.620 | 0.019 | 53.5 |
| Level 1.3 B  | 2  | 1.872 | 0.011 | -0.236 | 0.006 | 0.561 | 0.022 | 82.4 |
| Level 1.3 C  | 2  | 3.456 | 0.071 | 0.030 | 0.028 | 0.689 | 0.007 | 27.1 |
| Level 1.3 D  | 2  | 4.020 | 0.039 | -0.212 | 0.008 | 0.689 | 0.006 | 27.1 |
| Level 1.3 E  | 1  | 3.366 | 0.147 | 0.688 | 27.4 |
| Level 1.3 F  | 1  | 3.039 | 0.033 | 0.685 | 28.2 |
| Level 1.4 A  | 2  | 3.273 | 0.022 | -0.028 | 0.085 | 0.706 | 0.026 | 21.3 |
| Level 1.4 B  | 2  | 1.645 | 0.040 | -0.482 | 0.028 | 0.634 | 0.005 | 47.7 |
| Level 1.4 C  | 2  | 3.290 | 0.002 | -0.028 | 0.014 | 0.544 | 0.021 | 92.7 |
| Level 1.4 D  | 2  | 4.260 | 0.034 | -0.516 | 0.001 | 0.586 | 0.004 | 69.3 |
| Level 1.4 E  | 2  | 4.084 | 0.013 | -0.495 | 0.048 | 0.657 | 0.003 | 38.4 |
| Level 1.4 F  | 1  | 3.974 | 0.178 | 0.672 | 32.9 |
Heated. At temperatures below 150°C, the rate of $\Delta^{47}$ resetting in shells, or (iii) a single cooking technique wherein shells are unevenly cooking techniques resulting in different thermal histories for different constituents. This could be a result of one of several scenarios including (i) the discarding of uncooked shells, (ii) the use of different cooking techniques for different shells, or (iii) a single cooking technique wherein shells are unevenly heated. At temperatures below 150°C, the rate of $\Delta^{47}$ resetting in biogenic aragonite necessitates that the duration of heating far exceeds a hypothetical 10-min cooking time (10). Cooking methods involving boiling are expected to require hundreds of hours to significantly affect $\Delta^{47}$ values (10) and, thus, would be indistinguishable from the control group shells. The uneven distribution of $\Delta^{47}$ values between valves, where approximately half of the shells remain unchanged, could be produced using a single cooking technique, which causes uneven heating of valves during cooking.

The evidence provided from clumped isotopes indicates that approximately 50% of P. pectinatus valves at the Cabo Rojo site in Puerto Rico were heated to temperatures well above that of boiling water, with no significant variance with respect to burial depth. The precise reason for this nonuniformity in $\Delta^{47}$ values, however, cannot be determined with the available data. Our findings could indicate either two separate preparation processes (or the preparation of only half the valves) or a single process that resulted in uneven heating within the specimen. Nevertheless, it can be clearly demonstrated that most of the shells were heated to temperatures greater than 100°C but no greater than 200°C. Future work implementing the clumped isotope technique in the study of ancient culinary practices would benefit from additional experimentation using different species and culinary preparation techniques.

Fig. 3. $\Delta^{47}$ values for uncooked modern shells and shell midden constituents from CRNWR and experimental results for cooked bivalves presented by Müller et al. (9). (A) $\Delta^{47}$ values of all modern and ancient samples measured in this study (black dots); colored regions display the probability distribution for each group of analyses. Shaded regions behind points delineate sample frequency and are generated using distriPlot for MATLAB by J. Dom. The location, estimated age ±2σ, and sample ID of radiocarbon measurements are shown in the bottom of the figure as stars. (B) $\Delta^{47}$ values (y axis) and temperature of heating (x axis) of Müller et al. (9) cooked clams. T½ estimates for $\Delta^{47}$ equilibration half-lives based on the Arrhenius model extrapolating through direct heating experiments of biogenic aragonite between 125° and 175°C (10). The secondary y axis for both figures is estimated maximum cooking temperature.

(Materials and Methods) CRNWR_P13 is a 2500-m² shell midden site located within the boundaries of the Cabo Rojo National Wildlife Refuge in southwestern Puerto Rico (Fig. 1). As seen in Fig. 2, excavation of a 1 m × 1 m test unit within the site revealed two distinct soil strata (based on color/texture). Stratum I was an artifact-rich midden deposit, composed of 10YR 3/3 loamy sand, which extended from the present soil surface to a depth of 35 to 45 centimeters below surface (cmbs). Artifacts were present from the outset (denoted by the “-” symbol in Fig. 2), but reached peak density at 15 to 30 cmbs (denoted by “X” in Fig. 2), and decreased thereafter to the end of Stratum I. For purposes of stratigraphic control, Stratum I was excavated as four vertically successive 10-cm levels. Two radiocarbon dates [Beta-492522 (2240 ± 30 bp) and Beta-492527 (2760 ± 30 bp)] were obtained from Stratum I, at the top and bottom of the shell midden deposit, respectively. Calibration of these dates using Calib v7.1 (http://calib.org, accessed 20 March 2019), the Marine13 Curve (27), and a local deltaR of −27 ± 24 yields ranges (±2σ) of 30 BCE to 180 CE for Beta-492522 and 730 to 430 BCE for Beta-492527. Stratum II consisted of a 10YR 4/4 clay loam and was almost entirely sterile (with some isolated artifacts having been shifted into this stratum as a result of land-crab burrowing). While bioturbation undoubtedly had occurred, only a minute number of artifacts appear to have been vertically displaced (with only 41 g of shell in Stratum II, as compared with nearly 20 kg in Stratum I). This fact, combined with the overall compactness and robusticity of the midden, as well as the as-expected vertical succession of radiocarbon dates, gives an overall impression of a robust and largely intact midden profile. Products of excavation include an abundance of marine shell (primarily bivalves) and smaller quantities of faunal bone and lithic artifacts. To date, no ceramic artifacts have been encountered at this site.

In total, nearly 20 kg of shell was recovered from the test unit. Shells used in the present analysis were removed from the four 10-cm levels of Stratum I (referred in text and figures as level 1.1 through level 1.4). In total, six randomly selected shells were analyzed from each of these four levels and six additional shells were collected at

Staudigel et al., Sci. Adv. 2019; 5 : eaaw5447 27 November 2019
the modern coastline (50 to 100 m distant from the midden). Selection of shells from successive levels was carried out not as a means of testing changes in cooking technique over time but in an attempt to improve the representativeness of the sample. While the number of samples is small relative to the size of the assemblage, the stratified random sampling approach used was judged as the most effective means of providing for a representative but still manageable number of samples. Bivalves were ground using a ceramic ball-mill grinder into a fine powder and stored in glass vessels. Approximately 50 mg of this powder was made into a water slurry and mounted to 1-cm glass slides for XRD analysis. The calculation of calcite content using the XRD results is discussed further in the Supplementary Materials.

Isotopic analysis was conducted following the methods described in previous manuscripts (9, 10, 25). Ten to twelve milligrams of carbonate powder were weighted into copper boats and placed in a manually actuated sample carousel on a vacuum line. Samples were dropped into the acid bath and reacted for 30 min; the liberated CO2 and water vapor was collected in a trap cooled using liquid nitrogen. Water-CO2 separation was facilitated by warming the trap to −90°C with a methanol slush. The CO2 was collected in a separate trap, and the residual water was then heated and pumped away. Following collection, the CO2 gas was passed through a column packed with Porapak Q beads and cooled between −20° and −30°C; this removes organic contaminants, which may produce isobaric interference in subsequent analysis. The CO2 was passed through a column packed with Porapak Q beads and equilibrated in a quartz vessel at 25° and 50°C and CO2 equilibrated in a quartz vessel at 100°C were used as reference gas to correct data into the absolute reference frame (24). Stochastic distributions were calculated using the Brand parameters for PDB as recommended by Daëron et al. (30). Replicate analyses of carbonate reference materials used for interlaboratory comparison (ETH 1, 2, 3, and 4) (31) yielded Δ17 values of 0.288 ± 0.009, 0.296 ± 0.010, 0.683 ± 0.014, and 0.538 ± 0.009, respectively, in the absolute reference frame. Analyses by Schauer et al. (32) using acid digestion at 90°C yielded Δ17 values 0.287, 0.280, 0.694, and 0.553‰, respectively, all of which fall within the uncertainty of our measurements of the same materials. Individual bivalve and ETH standard analyses are presented in data S1. The isotopic data for all bivalves, including paleotemperature estimates, are summarized in Table 1.

The calibration for estimating peak heating temperatures was constructed using data from a previously published study (9), which showed a relationship between cooking temperatures and the Δ17 value of bivalve shells. These data largely agree with the dataset from a direct heating experiment of sclerosponge aragonite (10), suggesting that this effect is common in marine biogenic aragonite.

### Supplementary Materials

Supplementary material for this article is available at http://advances.sciencemag.org/cgi/content/full/5/11/eaaw5447/DC1

Supplementary discussion on quantifying mineralogy using XRD

---

**REFERENCES AND NOTES**

1. A. Dalby, *Food in the Ancient World from A to Z* (Routledge, 2013).
2. S. Grainger, The myth of Apicus. *Gastronomics* 7, 71–77 (2007).
3. D. Urem-Kotsou, K. Kotsakis, B. Stern, Defining function in Neolithic ceramics: The example of Makiyvalos, Greece. *Documenta Praehistorica* 29, 109–118 (2002).
4. C. Shafer-Elliott, *Food in Ancient Judah: Domestic Cooking in the Time of the Hebrew Bible* (Routledge, 2014).
5. L.K. Horsley, R.B. Walker, K.D. Hollenbach, What’s for dinner? Investigating food-processing technologies at Dust Cave, Alabama. *Southeastern Archaeology* 29, 182–190 (2010).
6. J.J. Parnell, R.E. Terry, P. Sheets, Soil chemical analysis of ancient activities in Cerén, El Salvador: A case study of a rapidly abandoned site. *Lat. Am. Antiq.* 13, 331–342 (2002).
7. S.M. Colley, The analysis and interpretation of archaeological fish remains. *Adv. Archaeol. Method Theory* 2, 207–253 (1990).
8. J.J. Parnell, R.E. Terry, Z. Nielsen, Soil chemical analysis applied as an interpretive tool for ancient human activities in Piedras Negras, Guatemala. *J. Archaeol. Sci.* 29, 379–404 (2002).
9. P. Müller, P.T. Staudigel, S.T. Murray, R. Vernet, J.-P. Barusseau, H. Westphal, P.K. Swart, Prehistoric cooking versus accurate palaeotemperature records in shell midden constituents. *Sci. Rep.* 7, 35557 (2017).
10. P.T. Staudigel, P.K. Swart, Isotopic behavior during the aragonite-calcite transition: Implications for sample preparation and proxy interpretation. *Chem. Geol.* 442, 130–138 (2016).
11. W. Stahl, R. Jordan, General considerations on isotopic paleotemperature determinations and analyses on Jurassic ammonites. *Earth Planet. Sci. Lett.* 6, 173–178 (1969).
12. Y. Guo, W. Deng, G. Wei, Kinetic effects during the experimental transition of aragonite to calcite in aqueous solution: Insights from clumped and oxygen isotope signatures. *Graeco. Cosmochim. Acta* 248, 210–230 (2019).
13. C. Ayés Suárez, Evaluación arqueológica tipo Fase 2, Angostura, Florida Amefera, Barceloneta, Puerto Rico (Consejo para la Protección del Patrimonio Arqueológico Terrestre de Puerto Rico, San Juan, 1988).
14. C. Ayés Suárez, Angostura: Un campamento Arcaico temprano del Vallee del Manatunabú. *Revista Universidad de America* 1, 24–37 (1989).
15. C. Ayés Suárez, O. Dávila Dávila, Angostura: Un campamento arcaico temprano del vallee del Manatunabú Bo, Florida Amefera, Barceloneta, Puerto Rico, (Consejo para la Protección del Patrimonio Arqueológico Terrestre de Puerto Rico, 1993).
16. M. Rodríguez López, En Ocho Trabajos de Investigación Arqueológica en Puerto Rico: Segundo Encuentro de Investigadores, J. A. Rivera Fontán, Ed. (Instituto de Cultura Puertorriqueña, 1997), pp. 17–30.
17. M. Rodríguez López, Excavaciones en yacimiento Arcaico de Manuca, Ponce, Puerto Rico: Informe Final (Consejo para la Protección del Patrimonio Arqueológico Terrestre de Puerto Rico, 2004).
18. I.C. Rivera-Collazo, Of shell and sand: Coastal habitat availability and human foraging strategies at Punta Candelero (Humacao, Puerto Rico). *Puertorriquena* 31, 272–284 (2010).
19. R.R. Ramos, E. Babilonia, L.A. Curet, J. Ulloa, The pre-Arawak pottery horizon in the Antilles: A new approximation. *Lat. Am. Antiq.* 19, 47–63 (2008).
20. W. F. Keegan, Archaic influences in the origins and development of taiño societies. *Cam. J. Sci.* 42, 1–10 (2006).
21. J. Ulloa Hung, Approaches to early ceramics in the Caribbean: Between unilineality and unilineality, in *Dialogues in Cuban Archaeology*, L. Ulloa, Eds. (University of Alabama Press, 2005), pp. 103–124.
22. P.K. Swart, N.P. James, D. Mallinson, M.J. Malone, H. Matsuda, T. Simo, Data Report: Carbonate Mineralogy of Sites Drilled during Leg 182, in *Proceedings of the Ocean Drilling Program Scientific Results*, A. C. Hine, D. A. Feary, M. J. Malone, Eds. (ODP, 2002).
23. K.J. Dennis, H.P. Affek, B.H. Passey, D.P. Schrag, J.M. Eiler, Defining an absolute reference frame for ‘clumped’ isotope studies of CO2. *Graeco. Cosmochim. Acta* 75, 7117–7131 (2011).
25. P. T. Staudigel, S. Murray, D. P. Dunham, T. D. Frank, C. R. Fielding, P. K. Swart, Cryogenic brines as diagenetic fluids: Reconstructing the diagenetic history of the Victoria Land Basin using clumped isotopes. Geochim. Cosmochim. Acta 224, 154–170 (2018).

26. P. J. Reimer, E. Bard, A. Bayliss, J. W. Beck, P. G. Blackwell, C. B. Ramsey, C. E. Buck, H. Cheng, R. L. Edwards, M. Friedrich, P. M. Grootes, T. P. Guilderson, H. Hafidison, I. Hajdas, C. Hatté, T. J. Heaton, D. L. Hoffmann, A. G. Hogg, K. A. Hughen, K. F. Kaiser, B. Kromer, S. W. Manning, M. Niu, R. W. Reimer, D. A. Richards, E. M. Scott, J. R. Southon, R. A. Staff, C. S. M. Turney, J. van der Plicht, IntCal13 and Marine13 radiocarbon age calibration curves 0–50,000 years cal BP. Radiocarbon 55, 1869–1887 (2013).

27. B. He, G. A. Olack, A. S. Colman, Pressure baseline correction and high-precision CO₂ clumped-isotope (Δ 47 ) measurements in bellows and micro-volume modes. Rapid Commun. Mass Spectrom. 26, 2837–2853 (2012).

28. K. W. Huntington, J. M. Eiler, H. P. Affek, W. Guo, M. Bonifacie, L. Y. Yeung, N. Thiagarajan, B. Passey, A. Tripati, M. Daëron, R. Came, Methods and limitations of ‘clumped’ CO₂ isotope (Δ 47 ) analysis by gas-source isotope ratio mass spectrometry. J. Mass Spectrom. 44, 1318–1329 (2009).

29. M. Daëron, D. Blamart, M. Peral, H. P. Affek, Absolute isotopic abundance ratios and the accuracy of Δ 47 measurements. Chem. Geol. 442, 83–96 (2016).

30. A. N. Meckler, M. Ziegler, M. I. Millán, S. F. Breitenbach, S. M. Bernasconi, Long-term performance of the Kiel carbonate device with a new correction scheme for clumped isotope measurements. Rapid Commun. Mass Spectrom. 28, 1705–1715 (2014).

31. A. J. Schauer, J. Kelson, C. Saenger, K. W. Huntington, Choice of 17O correction affects clumped isotope (Δ 47 ) values of CO₂ measured with mass spectrometry. Rapid Commun. Mass Spectrom. 30, 2607–2616 (2016).

Acknowledgments: Archaeological investigations were conducted under the terms of ARPA Permit CRJNWR121517, issued by the U.S. Fish and Wildlife Service. Funding: The Thermo-253 mass spectrometer was acquired using funds from NSF grant EAR0926503 to P.K.S. Author contributions: Samples were collected from the Cabo Rojo Wildlife Refuge by A.P., C.A.L.-D., and W.J.P. Analyses were conducted by P.T.S. in the laboratory of and under the supervision of P.K.S. Manuscript was prepared by P.T.S. and revised by co-authors. Competing interests: The authors declare that they have no competing interests. Data and materials availability: All data needed to evaluate the conclusions in the paper are present in the paper and/or the Supplementary Materials. Additional data related to this paper may be requested from the authors.

Submitted 3 January 2019
Accepted 18 September 2019
Published 27 November 2019
10.1126/sciadv.aaw5447

Citation: P. T. Staudigel, P. K. Swart, A. Pourmand, C. A. Laguer-Díaz, W. J. Pestle, Boiled or roasted? Bivalve cooking methods of early Puerto Ricans elucidated using clumped isotopes. Sci. Adv. 5, eaaw5447 (2019).
Boiled or roasted? Bivalve cooking methods of early Puerto Ricans elucidated using clumped isotopes
Philip T. Staudigel, Peter K. Swart, Ali Pourmand, Carmen A. Laguer-Díaz and William J. Pestle

Sci Adv 5 (11), eaaw5447.
DOI: 10.1126/sciadv.aaw5447