ALTERNATIVE EXPLANATIONS FOR ANOMALOUS ¹⁴C AGES ON HUMAN SKELETONS ASSOCIATED WITH THE 612 BCE DESTRUCTION OF NINEVEH

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ABSTRACT. Three factors—contamination, a dietary reservoir effect, and a regional Δ^{14} C anomaly—are considered as possible contributing explanations for an almost 2-century offset between the historically documented age of 612 BCE and the calibrated ages of 9¹⁴C determinations obtained on 3 human skeletons directly associated stratigraphically with an archaeologically—and historically—defined 612 BCE event at the ancient site of Nineveh in northern Mesopotamia (Iraq). We note that on the order of a 1% (~80 yr) offset caused by one or a combination of these 3 factors, or other as yet unidentified additional factor(s), would be sufficient to move the average measured ¹⁴C age of these bone samples within the major "warp" in the ¹⁴C timescale during the mid-1st millennium BCE. We provide what we believe to be sufficient evidence that contamination is not a major factor in the case of these bone samples. At this time, we lack appropriate data to determine with sufficient rigor the degree to which a dietary reservoir effect may be contributing to the offset. At present, a posited regional Δ^{14} C anomaly does not appear to be supported on the basis of data from several other localities in the Near East of similar age. One purpose of presenting this data set is to solicit comparisons with ¹⁴C values obtained on samples from additional, historically well-documented, known-age archaeological contexts for this time period in this and adjacent regions.

INTRODUCTION

In the late 8th and 7th centuries BCE, ancient Nineveh (Akkadian: *Ninua*) was the most populous city of the Neo-Assyrian Empire. During most of the period of its existence, this empire was the most militarily and economically powerful political entity in Western Asia. At its maximum geographic extent, it exercised direct rule or had undisputed hegemony over most of Mesopotamia, parts of southern Anatolia, the whole of the Levant and, for part of that period, Lower and Middle Egypt (Bury et al. 1954). However, beginning not later than the fourth decade of the 7th century BCE, serious external challenges, as well as various internal difficulties including civil strife, began to reduce the legendary prowess of its military forces.

Late in that century, a coalition that included large contingents of Babylonians and Medes, attacked, captured, and sacked Nineveh (Oates 1965; Grayson 1975; Brown 1999). This event was not only noted in contemporary Babylonian documents, but also in allusions that came to be included in the ancient Hebrew religious canon. The event was also marked in the much later writings of a number of Greco-Roman authors.

Today, the remains of this city comprise an area of approximately 750 hectares of essentially continuous ruins enclosed within the remnants of ancient stone and mud-brick walls that include 15 external gates. A long series of excavations, beginning in the mid-19th century, have been undertaken by various investigators. The excavations have revealed extensive architectural and cultural remains as well as physical evidence of the above-mentioned destruction, which included unburied skeletons of the last defenders (Stronach 1997; Pickworth 2005).

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STRATIGRAPHIC AND ARCHAEOLOGICAL CONTEXTS

Up until the second decade of the 20th century, there had been a general understanding among Assyrologists that the fall of Nineveh had taken place in 606 BCE. The placement of the destruction event in 612 BCE was established in the early 1920s with the discovery in the collections of the British Museum of a cuneiform tablet [BM 21, 901], which describes events that took place between the 10th (616–615 BCE) and 17th (609–608 BCE) years of the reign of the Babylonian King Nabopolassar (Akkadian: *Nabû-apal-usur*). This text states that Nineveh was taken in the month $\hat{A}bu$ of the 14th year of his reign or July/August, 612 BCE. Since that discovery, the 612 BCE date for the destruction of Neo-Assyrian Nineveh has been regarded as securely documented (Gadd 1923:3).

The human skeletal remains used in this study were collected in 1990 during archaeological excavations conducted under the direction of one of us (DS) at the Halzi Gate (Figure 1), the most southerly bastion on the long eastern city wall of Nineveh (Stronach 1997; Pickworth 2005). Of the 15 external gates known to have been constructed by the Assyrian King Sennacherib (704–681 BCE) at the time he enlarged Nineveh, the Halzi Gate represents 1 of the 2 most heavily fortified portals. This has led to a conjecture that the most southerly zone of Nineveh represented the most militarized section of the ancient city (Lumsden 1998:818, Figure 9). One consequence of its location so far from the center of the city was that no subsequent effort was made to reoccupy this outlying area following the 612 BCE destruction event.



Figure 1 View of Halzi Gate from the east. The human skeletal remains used in this study had not yet been exposed. They were subsequently recovered from an inner portion of the narrow central passage. Vertical rod scale = 2 m in 50 cm units. Horizontal rod scale = 1 m in 10 cm units. Photograph courtesy of D Stronach.

All human skeletal samples used in this study were excavated from the central passageway of the Halzi Gate (Figure 1). This single entryway was flanked on each side by 3 separate towers. These towers were originally built of fine ashlar blocks and each tower was crowned by crenellated stones. Fronting the gate was a moat that was fed from the River Khosr. Clearly, the forgoing combination of moat, gate, walls, and bastions represents a highly defended space (Stronach 1997). However, evidence of the declining resources of the Assyrian state may be reflected in the inferior workmanship of the repairs to 2 of the towers that had clearly been damaged in a slightly earlier, otherwise

unsuccessful attack. Further indications of declining security are evidenced by a blocking of mudbrick that narrowed the central corridor of the gate.

The disposition of the 12 excavated skeletons, all tightly concentrated in the central narrowed passageway, makes it clear that they died in the same event under dramatic circumstances (Figure 2). Interspersed amongst the skeletal material were multiple socketed trilobate arrowheads and an iron spearhead. The skeleton of a horse killed in the melee was found immediately outside the outer entrance of the passageway. Amongst the skeletons were various personal possessions and one of these, a blue chalcedony conoid stamp seal, is of particular interest. The iconography of the seal



Figure 2 Overhead view of part of the gate passage showing skeleton S12 *in situ* on pavement. Photograph courtesy of D Pickworth.

(NIN 90/25) is distinctly Babylonian from the 7th century BCE and does not occur earlier (Pick-worth 2005:313, Figures 40, 41). The pottery smashed on the floor of the passageway is also of late Neo-Assyrian date. The bodies were probably covered almost immediately by fallen mud-brick from the crumbling roof of the passage, and this overburden then remained undisturbed until the time of the UC Berkeley excavations in this specific area in 1990. An osteological analysis of the bones indicates that a number of these individuals had suffered injuries in earlier battles and had specifically developed musculature from the practice of archery (E Barnes, personal communication, 2005). On the basis of the archaeological evidence and historical data, it is clear that these skeletal remains can be associated with a specific event that occurred in the summer of 612 BCE.

AMS ¹⁴C, STABLE ISOTOPE (δ^{13} C, δ^{15} N), AND C/N DATA

Table 1 lists AMS-based ¹⁴C measurements obtained on ultrafiltered (>30 kD) gelatin prepared using procedures based on Brown et al. (1988) as described in greater detail in Beaumont et al. (2010). These measurements were obtained from 9 human bone samples taken from 3 skeletons associated with the 612 BCE destruction level at the Halzi Gate, Nineveh. Bone samples from 12 skeletons from the Halzi Gate area were made available for examination. While procedures to isolate ultrafiltered gelatin were applied to bone samples from all 12 skeletons, sufficient protein yields were obtained on bone from only 3 of the skeletons.

Table 1 ¹⁴C determinations on human skeletons excavated from the 612 BCE destruction level at the Halzi Gate, Nineveh. Single interval 2- σ range calibration values expressed for intercepts >0.95 relative area under probability distribution. Multiple intercepts listed for each intercept with relative areas under probability distributions >0.10 noted in parentheses.^a

	Calibrated age	Multiple intercepts relative
¹⁴ C age (yr BP)	2-σ range	area probability distribution
$2665\pm20 \text{ BP}$	835–795 cal BCE	
$2575\pm15~BP$	800–765 cal BCE	
$2575\pm20 \text{ BP}$	800–765 cal BCE	
$2540\pm20~BP$	795–750 cal BCE	(0.52)
	690–665 cal BCE	(0.22)
	645–590 cal BCE	(0.21)
$2565\pm25~BP$	805–750 cal BCE	(0.79)
	685–665 cal BCE	(0.13)
$2585\pm25~BP$	810-760 cal BCE	
$2585\pm25~BP$	810-760 cal BCE	
$2595\pm25~BP$	810–765 cal BCE	
$2645\pm30\text{ BP}$	845–785 cal BCE	
	$2575 \pm 15 \text{ BP}$ $2575 \pm 20 \text{ BP}$ $2540 \pm 20 \text{ BP}$ $2565 \pm 25 \text{ BP}$ $2585 \pm 25 \text{ BP}$ $2585 \pm 25 \text{ BP}$ $2595 \pm 25 \text{ BP}$	14 C age (yr BP)2-σ range2665 ± 20 BP 2575 ± 15 BP835–795 cal BCE 800–765 cal BCE2575 ± 20 BP800–765 cal BCE2540 ± 20 BP795–750 cal BCE 690–665 cal BCE 645–590 cal BCE2565 ± 25 BP 2585 ± 25 BP 2585 ± 25 BP 2595 ± 25 BP810–760 cal BCE 810–760 cal BCE 810–765 cal BCE

^aCalibration using CALIB 5.0.2 (Stuiver and Reimer 1993) and IntCal04 data set (Reimer et al. 2004).

^bA third ¹⁴C measurement on a bone from skeleton S6 yielded a value of 2840 ± 25 BP (UCIAMS-41649). Because this value was obtained on trace amounts of ultrafiltered gelatin, it has not been included in this discussion.

Based on the amount of ultrafiltered gelatin product obtained from a similar weight of bone, it was determined that bone from Halzi Gate human skeleton S10 was the best preserved from a biogeochemical perspective, and thus 6¹⁴C measurements were obtained on different bones from this skeleton. In addition, 3 ¹⁴C measurements were obtained on human skeleton S6 and 1 ¹⁴C determination was obtained from skeleton S8. AMS-based ¹⁴C measurements were obtained at the Keck Carbon Cycle AMS Laboratory, Department of Earth System Science, University of California, Irvine (Southon et al. 2004) and are expressed as conventional ¹⁴C values (Stuiver and Polach 1977).

Calibration of the ¹⁴C age for each measurement utilized CALIB 5.0.2 program protocols (Stuiver and Reimer 1993) employing the IntCal04 data set (Reimer et al. 2004). In Table 1, single interval 2- σ range calibration values are expressed for intercepts representing >0.95 of the relative area under the probability distribution. In cases of multiple intercepts, the 2- σ ranges with relative areas under probability distributions of >0.10 are noted in parentheses.

Table 2 lists δ^{15} N, δ^{13} C, and C/N ratios measured on ultrafiltered gelatin from bone and teeth (primarily dentin) samples from skeleton S10. Stable isotope values were obtained on a continuousflow stable isotope ratio mass spectrometer (Delta-Plus CFIRMS) interfaced with a Fisons NA-1500.

	δ^{15} N	$\delta^{13}C$	Ν	С	C/N		
Sample nr ^a	(‰)	(‰)	(%)	(%)	(wt%)		
UCIAMS-42515a	10.8	-19.2	15.6	43.2	2.8		
UCIAMS-42516a	11.1	-18.9	15.8	43.9	2.8		
UCIAMS-28521b	9.4	-19.4	14.7	41.4	2.8		
UCIAMS-28522b	8.4	-20.0	13.2	37.0	2.8		

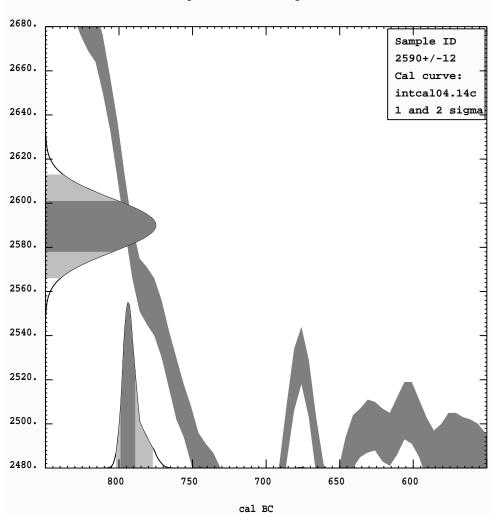
Table 2 Stable isotope and C/N ratios on ultrafiltered (>30,000 MW) gelatin from bone and teeth from Halzi Gate human skeleton S10.

 $^{a}a = \text{bone}; b = \text{teeth}.$

APPARENT ANOMOLY AND ALTERNATIVE EXPLANATIONS

This study assumes that the individuals from which our bone samples have been derived ceased to be part of their active terrestrial carbon exchange reservoir (i.e. died) within ± 1 yr of 612 BCE. The calibrated value of the mean age of the ¹⁴C values obtained on these 9 bone samples (Table 1) as illustrated in Figure 3 yields an indicated age a little less than 2 centuries too old. To account for this known-age/calibrated ¹⁴C age offset in the Halzi Gate human skeletal measurements, a number of factors could conceivably be responsible. Some of these factors would cause shifts in the indicated ages but, in our view, would clearly be of insufficient magnitude to account for the observed anomaly. For example, studies of turnover times for the organic carbon pool in human bone are of the order of 2 to 3 decades for young adults (Waterlow et al. 1978; Wild et al. 2000; Hedges et al. 2007); therefore, this cannot account for more than a small fraction of the apparent discrepancy.

We here consider 3 factors that have the capacity to cause all or a substantial part of the observed anomaly: sample contamination, a dietary reservoir effect, and a regional atmospheric Δ^{14} C offset. As illustrated in Figure 4, whatever factor or set of factors may be responsible, an anomaly would have to be only a little more than -1%, or approximately -80 yr, to move the measured mean 14 C ages of these skeletons onto a 300-yr "time warp" in the 14 C timescale in the mid-1st millennium BCE. This "wiggle" in the 14 C timescale, labeled as de Vries effect offset IIIb in Taylor et al. (1996) based on data from Stuiver and Reimer (1993: Figures 3A–D), represents the largest 14 C timescale excursion of the middle and late Holocene.

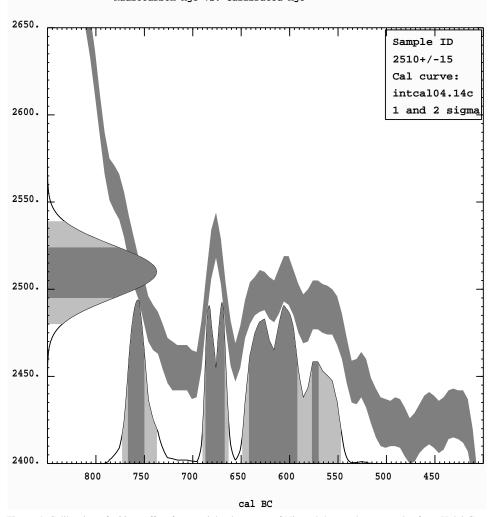


Radiocarbon Age vs. Calibrated Age

Figure 3 Calibration of weighted average of Nineveh human bone samples (n = 9) from Halzi Gate [2590 ± 12 BP]. Plot using CALIB 5.0.2 (Stuiver and Reimer 1993) employing the IntCal04 data set (Reimer et al. 2004).

DISCUSSION

With regard to the possibility of sample contamination, we are very cognizant of the decades-long history of the challenges involved in obtaining accurate ¹⁴C age determinations on bone samples (Taylor 1987:53–61; 1992; Stafford 1984; Bronk Ramsey et al. 2004; Beaumont et al. 2010). There are, of course, several biogeochemical issues that have the capability to cause anomalous ages to be obtained on our bone samples. The first relates to the well-known problem of bone seriously depleted in collagen content. The second involves the failure of a given chemical pretreatment protocol to remove essentially all, or all but for a few percent, of the non-autochthonous organics from bone samples. As noted previously, the >30-kD percentage yields from all but one of our bone samples on which ¹⁴C values were obtained indicated that they had retained a significant fraction of their principle protein constituent, collagen. C/N ratios of 2.8–3.0 (wt%) are typically associated with



Radiocarbon Age vs. Calibrated Age

Figure 4 Calibration of -80-yr offset from weighted average of Nineveh human bone samples from Halzi Gate [2540 \pm 12 BP]. Plot using CALIB 5.0.2 (Stuiver and Reimer 1993) employing the IntCal04 data set (Reimer et al. 2004).

Holocene-age bones that have retained >10% amounts of intact collagen. (Ratios in the 3 to 3.5 range are noted when reported as molar ratios.) The C/N ratios obtained on two of our samples (Table 2) are reported as wt%. (As noted in footnote b in Table 1, one of the bones that yielded a value of 2840 ± 25 BP (UCIAMS-41649) was obtained on trace amounts of ultrafiltered gelatin. Because of that, it has not been included in this discussion.)

To monitor possible contamination of these bone samples during pretreatment, we employed infinite ${}^{14}C$ age (>70 kyr) bone as chemistry blanks and known-age bone standards whose ultrafiltered gelatin were prepared at the same time as our unknown age samples. Evidence of contamination was observed neither in ${}^{14}C$ ages of the sample blanks nor in the ${}^{14}C$ ages of known-age bone. On this basis, our data does not support a view that the procedures employed to prepare the ultrafiltered gelatin introduced contamination into the samples or failed to remove a significant fraction of any non-

autochthonous organics from the bone. Based on the biogeochemical conditions of these bone samples and as a result of the application of our pretreatment protocols for bone ¹⁴C dating, it is our view that sample contamination by itself does not provide a sufficient explanation for the age offset observed. The only proviso that might be noted is that 2 ¹⁴C values obtained on skeleton S6 do not overlap at the 2- σ level. One explanation that might be posited is that sample pretreatment protocols in this case were not totally effective.

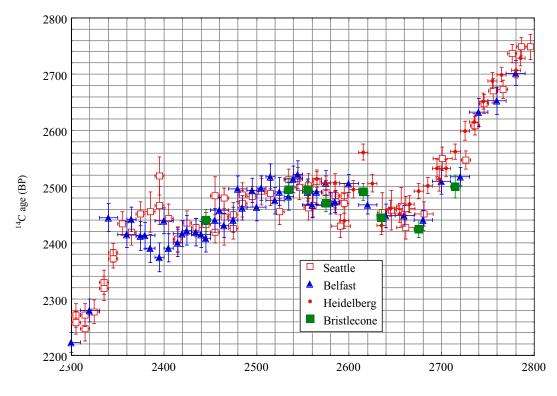
The second factor—a reservoir effect due to a significant amount of freshwater fish in the diet of the individuals from which the bone samples were obtained—is an option that requires additional study. We are aware of the significant age offsets that can occur due to the variable amounts of biomass (e.g. fish) ingested by organisms that are derived from localized segments of the carbon cycle (e.g. marine or freshwater environments) not in equilibrium with contemporary atmospheric ¹⁴C concentrations. Reservoir effects of significant magnitude have been reported for a number of regions, but the overwhelming majority of studies of reservoir effects have focused on marine environments. There are a relatively few number of examples of studies where mammalian bone from terrestrial environments have appeared to yield age offsets caused by the presence of marine or freshwater biomass in the diet of the subject individuals.

The most detailed study of which we are aware reported ¹⁴C and stable isotope study of skeletal samples recovered in archaeological sites situated adjacent to the Iron Gates Gorge region of the Danube River, which currently constitutes the international boundary between Serbia and Romania (Cook et al. 2001, 2002). Analysis of the faunal collections at these sites indicated that there had been substantial fish consumption by Mesolithic and early Neolithic Iron Gates human populations. A study that measured the ¹⁴C ages of collagen isolated from a set of 5 directly associated human and ungulate bone samples indicated that the human bone sample ages were consistently older, ranging from about 300 to 600 yr, with the weighed mean offset between paired samples being 440 \pm 45 yr. This research also obtained δ^{13} C and δ^{15} N measurements on each paired human/ungulate bone set to derive a relationship between the isotopic values and age offset values. Previously obtained stable isotope data on human bone samples from this region obtained by other investigators permitted the determination of the end points, i.e. 100% terrestrial diet to 100% freshwater aquatic diet, and thus an estimate of the linear relationship between δ^{15} N and age offset could be empirically calculated (Cook et al. 2001, 2002).

While the presence of freshwater fish in the diet of human populations can be responsible for shifting ¹⁴C values in bone samples, the potential significance of a reservoir effect due to fish consumption in the Nineveh population appears to be problematical based on data available from archaeological investigations undertaken in the region. Excavations conducted at various occupation sites situated in the upper reaches of the Tigris River have not reported the presence of significant amounts of the skeletal remains of fish. On the basis of current understandings, from at least the 6th millennium BCE onward, cultivated cereal grains and meat from domesticated animals constituted the major source of the food biomass for these populations.

An additional complication is that we are not aware of any published data measuring a sufficient number of δ^{13} C and δ^{15} N values on a similar data set from the any upper Tigris aquatic environmental regime that would permit us to estimate a linear isotopic/age offset relationship for our site. While we have obtained δ^{13} C and δ^{15} N values on ultrafiltered gelatin from bone (and teeth) from skeleton S10 (Table 2), we have not as yet been able to obtain a large sample of fish bone of similar age from this site to obtain comparable δ^{13} C and δ^{15} N values. We plan to obtain comparable data on *contemporary* fish bone samples taken from the Tigris in this general locality, but we recognize that significant changes in stable isotope values because of variations in the geochemical environment of the Tigris over the last 3 millennia may significantly limit the comparative value of this data. Therefore, we will also seek to measure ¹⁴C and δ^{13} C and δ^{15} N values in fish bone from other dated archaeological contexts.

The third major factor that might conceivably shift the calibrated values would involve the projected existence of a heretofore unrecognized regional Δ^{14} C anomaly for this part of Western Asia in this time period. As plotted in Figure 5, the Northern Hemisphere IntCal04 interpolated data set (Reimer et al. 2004) for the mid-1st millennium BCE (2300 to 2800 cal BP = 350 to 850 cal BCE) is based on high-precision ¹⁴C age determinations from 3 ¹⁴C laboratories on tree-ring dated wood samples. Eight additional data points have been plotted in Figure 5. These are heretofore unpublished ¹⁴C determinations measured by this laboratory on bristlecone pine wood. The dendrochronological ages of these samples were determined by C Wesley Ferguson (Laboratory of Tree Ring Research, University of Arizona), supplied to Hans Suess (La Jolla/Mt. Soladad Radiocarbon laboratory, University of California, San Diego), dated by his laboratory in the late 1960s, and now in the possession of one of us (RET). Detailed results of the ¹⁴C dating of these and other tree-ring dated bristle-cone pine samples from the 1st millennium BCE period will be reported elsewhere.



Calibrated age (BP)

Figure 5 Relationship between ¹⁴C (BP) and dendrochronological (cal BP) ages for the period 2300–2800 cal BP (350–850 cal BCE). The University of Washington (Seattle), Queen's University (Belfast), and Heidelberger Akademie der Wissenschaften (Heidelberg) data are those used to construct the IntCal04 interpolated data set for that time interval (Reimer et al. 2004). The bristlecone pine data is based on ¹⁴C measurements obtained at the Keck Carbon Cycle Accelerator Mass Spectrometry Laboratory, University of California, Irvine on wood samples whose dendrochronological age was determined by the late C Wesley Ferguson (Laboratory of Tree Ring Research, University of Arizona), supplied to the late Hans Suess (La Jolla/Mt. Soladad Radiocarbon laboratory, University of California, San Diego), dated by his laboratory in the late 1960s, and now being curated by one of us (RET).

We wish to emphasize that the existence of a Near Eastern 7th–6th century BCE regional Δ^{14} C anomaly is not supported by several existing data sets. For example, Bruins and van der Plicht (2005) have reported ¹⁴C values obtained on both charcoal and cereal grains from a level at the site of Tell el-Quderirat in northeastern Sinai associated with Babylonian military campaigns of either 602 or 600 BCE. These data provide no evidence of an offsetting Δ^{14} C signature at the end of the 6th century BCE in that region. In addition, Bruins (personal communication, 2010) reports that as yet unpublished ¹⁴C values associated with the 602 BCE destruction of the Philistine coastal site of Ashkelon by Babylonian forces shows no Δ^{14} C offset.

For the purposes of this study, we wish to note that a rapid, very short-term oscillation in the InCal04 calibration curve plot in the interval of 2605 and 2645 cal BP (centered on 2625 cal BP), which is documented in the data used in InCal04 by only one ¹⁴C/dendrochronological data point at 2625 cal BP, is slightly offset from the ¹⁴C value on Bristlecone pine wood sample of the same dendrochronological age that we have obtained. Clearly, this data has no direct bearing on the validity of the suggestion of a postulated regional Δ^{14} C anomaly except that, if confirmed, it slightly adjusts the amount of offset that would be necessary to lower the ages of the Nineveh bone samples into the major mid-1st millennium "warp" in the ¹⁴C timescale.

CONCLUSION

We have noted that an approximately -1% (-80 yr) younger age caused by one or more of the 3 offsetting factors considered, or one or more other, as yet, unidentified factor(s), could move the average measured ¹⁴C age of these bone samples within the major ~300-yr "warp" in the ¹⁴C timescale in the mid-1st millennium BCE. At this time, we are only able to present evidence supporting the view that sample contamination alone cannot account for the age offset. We are aware that the lack of fish bone in archaeological collections from this region and time period calls into question the suggestion that a dietary reservoir effect might be, at least in part, responsible the age offset. Also, there is no evidence for the presence of a posited regional Δ^{14} C anomaly in Western Asia at this time, and thus, we recognize that this is the least likely factor even as a partial explanation.

One purpose of presenting this data set is to solicit comparisons with ¹⁴C values obtained from additional historically documented known-age archaeological contexts for this time period in this and adjacent regions and solicit the collaboration of those possessing samples from secure depositional contexts from sites in this region. We plan to undertake additional ¹⁴C and stable isotope studies that will definitively affirm or exclude the possibility of a dietary-based offset for human bone in this region. Toward this end, we will seek to measure associated non-bone organic materials and skeletal samples of animals that are known not to have eaten fish from this time period and region to expand our data base. We also plan to obtain additional measurements on tree-ring dated bristlecone pine samples to further contribute to the suite of Northern Hemisphere calibration values documenting the ¹⁴C timescale for the middle portion of the 1st millennium BCE.

ACKNOWLEDGMENTS

We thank the Gabrielle O Vierra Memorial Fund for financial support (RET). We also wish to express our appreciation to Hendrik J Bruins for his helpful comments as well as pointing us to comparative ¹⁴C data contained in Bruins and van der Plicht (2005) as well as providing unpublished data. The information provided by Ethne Barnes, PhD to DS and DP regarding the significance of the musculature exhibited on the Nineveh bones and by Paul Giem, MD to RET regarding the possible existence of a ¹⁴C anomaly in bone samples from the 612 BCE destruction layer at Nineveh is also acknowledged.

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