LOIS J. SCHIFFER Assistant Attorney General

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ALLISON RUMSEY U.S. Department of Justice Environment & Natural Resources Div. Office of the Assistant Attorney General 950 Penn. Ave., N.W., Room 2740 Washington, DC 20530 (202)514-0750 (202) 305-2573 facsimile

KRISTINE OLSON United States Attorney TIMOTHY W. SIMMONS Assistant United States Attorney District of Oregon 1000 S.W. 3rd Ave., Ste 600 Portland, Oregon 97204-2902 (503) 727-1156 (503) 727-1117 facsimile tim.simmons@usdoj.gov OSB#92461

IN THE UNITED STATES DISTRICT COURT

FOR THE DISTRICT OF OREGON

ROBSON BONNICHSEN, et al.,)
) FEDERAL DEFENDANTS'
Plaintiffs,) NOTICE OF RADIOCARBON
) RESULTS AND NOTICE OF
V.) DETERMINATION THAT THE
) HUMAN REMAINS ARE
UNITED STATES OF AMERICA, et al.,) NATIVE AMERICAN
)
Defendants.)
)
) Civil No. 96-1481 JE

As required by the Court's oral order of September 14, 1999, the federal-defendants are submitting the final radiocarbon test results. In addition, as described below, the U.S. Department of the Interior has made the initial NAGPRA determination that the human remains at issue in this action are Native American.

A. <u>Radiocarbon Results</u>

The four radiocarbon dates of the samples from the metatarsal and left tibial crest have been completed by the Beta Analytic Labs, Inc., the University of Arizona, and the Radiocarbon Laboratory at University of California, Riverside. Two of the four dates are consistent with the 1996 radiocarbon date of the left fifth metacarpal submitted by Benton County. See Determination That The Kennewick Human Skeletal Remains are "Native American" for Purposes of the Native American Graves Protection and Repatriation Act (Exhibit A) at Attachment 1, Letter From Darden Hood, Beta Analytic, Inc., to Dr. Francis P. McManamon, October 17, 1999 and Attachment 3, Letter from R.E. Taylor, University of California at Riverside, to Dr. Francis P. McManamon, December 20, 1999. The Beta sample cate was reported as 8410 +/- 40 BP, a date almost identical to the 1996 Benton County date, and the University of California Riverside Sample 1b was reported as 8130 +/- 40 BP. Id. The samples from the tibial crest reported more recent, but still quite old, dates. See Letter From Douglas Donohue, University of Arizona, to Dr. Francis P. McManamon, January 10, 2000 at Attachment 4, and Taylor 1999 at Attachment 3. The University of Arizona laboratory dated the sample from the tibial crest as 5570 +/- 1000 BP. The University of California at Riverside reported the date of its tibial crest sample as 6940 +/- 30 BP. The more recent dates on the tibial crest samples

Page 2: 'Federal Defendants' Notice to Court of Radiocarbon Results And Of Native American Determination

suggest that "new carbon" from the burial environment entered and contaminated the tibia. <u>See</u> Determination That The Kennewick Human Skeletal Remains are "Native American" for Purposes of the Native American Graves Protection and Repatriation Act at p.4.

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Indeed, the carbon in all the samples was quite low and this low carbon may indicate that some of the samples were contaminated by more recent foreign carbon – not an unusual situation in human remains of this age. Taylor Attachment 3 at p.4.¹ The sample from the metacarpal submitted by Benton County in 1996 differs substantially from these samples in that the 1996 sample had a much higher carbon content in the bone sample. This difference may result from the fact that the metacarpal was found in intact sediments within the cranium; this may have protected it from the deterioration. See Statement of Dr. James Chatters, December 17, 1999 (Attachment 5); Taylor Attachment 3 at p.3.

Nevertheless, all of the analyses performed to date are consistent in showing that the remains are thousands of years old. Moreover, the Department of the Interior has a high degree of confidence that the results of the radiocarbon dating of the metacarpal and the metatarsal are accurate given a number of factors, including: (1) that the radiocarbon dates for both bones are almost identical (albeit tested with different levels of carbon); (2) that Beta has confidence in its date because of the good quality of the collagen of its metatarsal sample; and (3) the dates are consistent with the supporting data from the 1999 non-destructive scientific studies. <u>See</u> Determination That The Kennewick Human Skeletal Remains are "Native American" for

¹University of California at Riverside is undertaking a further analysis of the sample that may assist the U.S. Department of the Interior in determining the source of the contamination. Taylor Attachment 3 at p.4.

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Purposes of the Native American Graves Protection and Repatriation Act at 3-4; Final Report of Non-Destructive Testing of the Kennewick Remains, February 1999 (Attachment 6).

B. <u>Determination that the Human Remains Are Native American, As Defined</u> By NAGPRA.

With the completion of the radiocarbon dating, the Department of the Interior has determined that these human remains are "Native American" as defined by NAGPRA <u>See</u> Determination That The Kennewick Human Skeletal Remains are "Native American" for Purposes of the Native American Graves Protection and Repatriation Act at 6. As discussed in the determination memorandum, all of the scientific data collected on these human remains supports the conclusion that the human remains belong to a culture that pre-dated the arrival of Columbus. <u>Id.</u>

Dated this 12th day of January, 2000.

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Respectfully submitted,

LOIS J. SCHIFFER Assistant Attorney General

Allison Russes (T.S.)

ALLISON RUMSEY Office of the Assistant Attorney General Env. & Natural Res. Div. U.S. Dept. of Justice

KRISTINE OLSON, OSB 73254 United States Attorney

TIMOTHY W. SIMMONS Asst. United States Attorney OSB 92461 Of Attorneys for Federal Defendants

DOI 05812

Page 4: Federal Defendants' Notice to Court of Radiocarbon Results And Of Native American Determination

OF COUNSEL: Russell Petit Office of Chief Counsel Office of Chief of Engineers Washington, D.C.

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Rebecca Ransom Office of Counsel Northwest Division Army Corps. of Engineers Portland, Oregon Carla Mattix U.S. Department of Interior Office of Solicitor Division of Conservation and Wildlife 1849 C Street, NW, Room 6557 Washington, D.C.

DOI 05813

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CERTIFICATE OF SERVICE BY MAIL

I certify that I made service of the foregoing FEDERAL DEFENDANTS'

NOTICE OF RADIOCARBON RESULTS on the parties herein by faxing and depositing in the

United States mail at Portland, Oregon, on January 12, 2000 a copy thereof, enclosed in a

postage prepaid envelope, addressed to:

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Alan L. Schneider, Esq. 1437 S.W. Columbia Street, Suite 200 Portland, OR 97201

Paula Barran, Esq. BARRAN & LIEBMAN 601 S.W. Second Ave., Suite 2300 Portland, OR 97204-3159 Attorneys for Plaintiffs in No. 96-1481

Michael Clinton, Esq. 8601 S.E. Revenue Rd., Boring, OR 97009 Attorney for Plaintiffs in No. 96-1516

David J. Cummings, Esq. Nez Perce Office of Legal Counsel P.O. Box 305 Lapwai, ID 84540-0305

Dan Hester, Esq. Fredericks, Pelzyger, Hester & White, LLC 1075 South Boulder Road, Suite 305 Louisville, CO 80027

SUSAN MYERS Paralegal Specialist

DOI 05814

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Table 1: C14 Samples and Radiocarbon Dates from Kennewick Skeletal Remains

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Radiocarbon Lab/Sample Number	Radiocarbon Age	Calibrated Radio- carbon Age
Beta Analytical Inc.		
Beta-133993	8410 +/- 40 BP	cal BP 9510-9405
Sample Catalog #: CENWW.97.R.24(MTa Sample #: DOI 1a Portion of right first metatarsal	ı)	and car Br 9343-9320
University of California at Riverside Radiocarbon Laboratory		
UCR-3807/CAMS-60684 Sample Catalog #: CENWW.97.R.24(MTa Sample #: DOI1b Portion of right first metatarsal	8130 +/- 40 BP ¹ .)	
UCR-3806/CAMS-60683 Sample Catalog #: CENWW97.L.20b Sample #: DOI2b Portion of left tibial crest	6940 +/- 30 BP ¹	
UCR-3476/CAMS-29578 Sample #: APS-PS-01 5 th left metacarpal	8410 +/- 60 BP [original C14 date from	n 1996 analysis]
University of Arizona NSP-Arizona AMS Facility		
AA-34818 Sample Catalog #: CENWW.97.L.20b Sample #: DOI2a Portion of left tibial crest	5750 +/- 100 BP	

Reported by UC-R as "apparent C14 age"



United States Department of the Interior

NATIONAL PARK SERVICE 1849 C Street, N.W. Washington, D.C. 20240

IN REPLY REFER TO

Memorandum

To:	Assistant Secretary, Fish and Wildlife and Parks
Through:	Director
From:	Departmental Consulting Archeologist
Subject:	Determination That the Kennewick Human Skeletal Remains are "Native American" for the Purposes of the Native American Graves Protection and Repatriation Act (NAGPRA)

Background

The interagency agreement between the Department of the Army (DOA) and the Department of the Interior (DOI), signed in March, 1998, delegated responsibilities to the DOI for certain decisions related to the set of human skeletal remains recovered from land managed by the Corps of Engineers (COE) near Columbia Park, Kennewick, WA. The agreement calls for the DOI to investigate and resolve two basic issues. First, we must determine whether or not the remains meet the definition of "Native American" according to the definition in the Native American Graves Protection and Repatriation Act (NAGPRA), as interpreted by DOI. Second, if the remains are Native American, the DOI will determine their disposition under the requirements of NAGPRA.

This memorandum describes the basis for the determination of the first of these actions. that is, whether or not the Kennewick skeletal remains are considered "Native American". as defined by NAGPRA.

As defined in NAGPRA, "Native American" refers to human remains and cultural items relating to tribes, peoples, or cultures that resided within the area now encompassed by the United States prior to the historically documented arrival of European explorers, irrespective of when a particular group may have begun to reside in this area, irrespective of when a particular group may have begun to reside in this area, and, irrespective of whether some or all of these groups were or were not culturally affiliated or biologically related to present-day Indian tribes.

DOI 05816

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If this set of remains is found to fit within the category of "Native American," issues related to cultural affiliation will be highly relevant to how disposition of the remains should be accomplished. However, this will be a subsequent step in our assistance to the DOA and is not addressed further in this memorandum. We currently are investigating the possible cultural affiliation of these remains.

The Kennewick Skeletal Remains are "Native American" as Defined by NAGPRA

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We now have sufficient information to determine that these skeletal remains should be considered "Native American" as defined by NAGPRA. The results of recent radiocarbon dating of small samples of bone extracted from the remains were given significant weight in making this determination. This interpretation is supported by other analyses and information regarding the skeletal remains themselves, sedimentary analysis, lithic analysis, an earlier radiocarbon date on a bone recovered with the other remains, and geomorphologic analysis (summarized in McManamon 1999).

A series of radiocarbon dates now available from the Kennewick skeletal remains indicate a clearly pre-Columbian date for the remains (Table 1 and discussed below). It is reasonable to conclude that the human remains from Columbia Park in Kennewick, WA, are "Native American" as defined by the Native American Graves Protection and Repatriation Act.

A variety of additional scientific information support this chronological placement and determination. Geomorphologic and sedimentary investigations of the river bank near the discovery site (Wakeley et al. 1998; Huckleberry et al. 1998) indicate that sediment layers consistent with these dates exist in the alluvial terrace where we believe the remains were buried originally. The documentation, examination, and analysis of the skeletal remains themselves (Powell and Rose 1999) suggest a pre-Columbian context for the remains. Comparison of sediments adhering to the skeletal remains and sediments from the river bank profile are consistent with the skeletal remains having beer, buried in sediments stratigraphically dated pre-7000 BP (Huckleberry and Stein 1999). Information from the analysis of the lithic artifact lodged in the ilium of the skeletal remains also is consistent with an ancient date for the remains themselves (Fagan 1999). In all, information derived using the methods and techniques of archeology, geomorphology, physical anthropology, sedimentology, and other scientific disciplines support this determination

Our determination that the Kennewick skeletal remains are "Native American" is based upon the scientific information that we have available. As explained in subsequent sections, this a reasonable determination based upon such information now on hand.

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Summary of the Radiocarbon Results

Four C14 dates have been reported for the samples extracted by the Department of the Interior and Corps of Engineers in September, 1999. The samples have been processed and dated by Beta Analytical, Inc. (BA), of Miarni, Florida, the Radiocarbon Laboratory of the University of California, Riverside (UC-R), and the NSF-Arizona AMS Facility of the University of Arizona (UA). Two of the four new dates show a substantial conformance with the initial radiocarbon date of the portion of the metacarpal submitted by Benton County in 1996 (see Table 1). All the carbon samples showed very low carbon content and this has slowed the processing of the samples and extended the time required to develop our interpretation of the C14 dates.

The BA date (Beta-133993) gave a conventional radiocarbon age of 8410 ---- 40 BP (Hood 1999a and Attachment 1). The equivalent calibrated radiocarbon age (using the two sigma, 95% probability) in years BP is cal BP 9510 to 9405 and cal BP 9345 to 9320. The bone sample used for this date was approximately half of the right metatarsal, one of the load-bearing bones of the foot (Sample DOI 1a). Analysis and processing of the sample at Beta indicated that the amount of organic carbon remaining in the sample was very low. The Laboratory Director of BA, Mr. Darden Hood, reported that "the original weight of the bone was 9.1 grams. The amount of collagen extracted was 0.030 grams (30.0 mg). This relates to a percent concentration of 0.3%. The value is very low due to the high mineral content of the submitted bone. 9.5 mg. Of the collagen was used for the analysis. This provided us with 3.2 mg. of carbon. The percentage of carbon is then calculated as 33.7% carbon within the collagen (Hood 1999b and Attachment 2)." Mr. Hood also reported that "by our standards, the collagen extract looked free of intrusive elements...It was vitreous in texture and golden in color as expected. It was free of visible contamination or deterioration. However, this does not preclude the presence of secondary [i.e., intrusive] environmental proteins (Hood 1999c)."

The Radiocarbon Laboratory of the UC-R processed and dated two of the Kennewick bone samples (Taylor 1999 and Attachment 3). Like the BA sample, both of these were very low in carbon content. Due to the low carbon content and the lack of clear collagenlike characteristics of the extracted carbon, the dates were reported as "the apparent C14 ages" for each sample (see Table 1). One of the samples (Sample DOI 1b) was dated as 8130 +/- 40 BP (UCR-3806/CAMS-60684), slightly different from the BA date for Sample DOI 1a, but not inconsistent with it. These two samples, in fact, are from the same bone, the right first metatarsal.

Both of these dates (Beta-133993) and (UCR-3806/CAMS-60684) are consistent with the earlier C14 date obtained from a portion of the 5th left metacarpal (Taylor et al 1998). The BA date, in fact is almost identical to the first C14 date.

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The other UC-R date is also old, an apparent C14 age of 6940 ± 30 BP (UCR-3806/CAMS-60683), but more recent than the other dates. This sample (Sample DOI 2b) from the left tibial crest also is more deteriorated than Sample DOI 1b. Sample DOI 2b contains only 2.3% of the carbon relative to the UC-R modern bone standard while Sample DOI 1b contains 14.3% of the modern standard.

The UA laboratory dated the second subsample from the left tibial crest (Sample DOI 2a). The date they obtained is also old, 5570 ± 100 BP (AA34818). This date is more or less consistent with the UC-R 3806/CAMS-60683 date and together they suggest that exogenous "new carbon" is pronounced in the left tibia from which these two samples were taken. The UA laboratory also reported a low carbon content for Sample DOI 2a (Donahue 2000a and b and Attachment 4). They recorded a carbon yield of .05 %, that is, the final mass of carbon based upon the initial mass of the bone. UA's analysis of this level of carbon content was that they could not determine the source of the carbon, i.e., whether it was inherent or exogenous.

Low Carbon and Possibility of Intrusive Contamination

One problem with dating bone samples with low carbon is that exogenous or intrusive carbon may have infiltrated the bone and become mixed with the endogenous or inherent carbon. If treatment of the sample before dating is not able to remove the intrusive carbon, any date from the sample will be distorted by the intrusive carbon. In most cases, it is younger carbon that is intrusive, for example, carbon from plant roots, soil microorganisms, or humic organic compounds in the soil. Usually such sources of exogenous carbon post-date the death and burial of the bone being dated. The effect of such mixing of "new carbon" with the original carbon in the bone is to make the date of the bone appear more recent than the true date.

In the case at hand, this may be the reason for the date from Sample DOI 2b. Taylor suggested this in his report on the C14 dating of the samples done by UC-R. "One interpretation [of the difference between the original date and the dates from these samples] is that the age offsets reflect varying percentages of more recent and/or modern contamination in both UCR-3806 and UCR-3807, with the percentage contribution of contamination increasing as a function of the decreasing residual collagen protein content (Taylor 1999a:1-2)."

If the only probable risk of intrusion by exogenous carbon is from more recent cr modern carbon, as seems likely, the dates for the Kennewick bone samples indicate strongly that the remains definitely are pre-Columbian, and therefore "Native American" as defined by NAGPRA.

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In certain geomorphologic circumstances, bone can be infiltrated by older carbon. If such "old carbon" is not removed in treatment prior to dating, dates will be distorted by appearing older than the bone itself. The geomorphic context in which we believe the Kennewick skeleton was buried and rested for many centuries is unlikely to have been affected by such contamination. There appears not to be an accessible and likely source for such carbon. Limestone, a common source of old carbon, is not prevalent in the watershed. Nor has there been much of an opportunity for such intrusion to have occurred through groundwater immersion of the bone by old carbon saturated water (Huckleberry et al. 1998; Wakeley et al. 1998).

Difference with the 1996 C14 Sample

The low amounts of carbon detected in the DOI samples extracted from the right metatarsal and left tibia of the Kennewick remains differ substantially from the carbon content of the bone sample (portion of the fifth left metacarpal) submitted to the UC-R Archaeology Lab by the Benton County Coroner's office in August, 1996. The carbon content of this sample (UCR-3476/CAMS-29578) has been reported by UC-R as "...68.8% of our modern reference sample and the relative concentrations of arnino acids was similar to that observed in our modern bone standard...(Taylor et al. 1998:1171-1172)"

This discrepancy between the carbon content observed in the 1996 sample and the samples analyzed in 1999 calls into question the relationship of the earlier sample to the rest of the human remains. It is unexpected and unusual, although not impossible, for an individual human skeleton to exhibit widely different concentrations of collagen in bones from different parts of the body.

Prior to the detailed examination of the Kennewick human remains in February, 1999, reported by Powell and Rose (1999) there were questions concerning whether the skeletal elements collected during July and August, 1996, were from a single individual. Powell and Rose demonstrated that the remains obtained from the original collector by the Corps of Engineers and curated since September, 1996, by them indeed were from a single individual. Also arguing for these bones being from the same individual is the fact that three independent radiocarbon dates consistently show the bones to date between about 8000 and 8500 BP.

We have received a more detailed description by the archeologist who originally collected the remains in 1996 (Egan 2000). This information indicates that the bone used for the 1996 C14 date was similar to other bones in appearance and might have been better protected from long term deterioration. There appears to be a photograph of the bone fragment to compare with the other bones. We shall verify this information using the photograph as best we can.

Page C_EXHIBIT A

Conclusion

The chronological information needed to make the determination that the Kennewick skeletal remains are "Native American" as defined by NAGPRA has been provided by the additional C14 testing conducted by the Department of the Interior and three radiocarbon laboratories. All the dates obtained predate 6000 BP and are clearly pre-Columbian. Two of the dates match closely the C14 date obtained in 1996 on another bone fragment believed to be from the skeleton.

Results of the earlier documentation, examination, and analysis of the remains themselves, sediment analysis comparing the sediment on the bones with sediment from the soil profile near where they were recovered, analysis of the lithic point embedded in the left ilium of the remains, and geomorphologic studies near the discovery site also support this determination.

 $\frac{1}{(\text{date})}$ Concur: Donald J. Barry, Assistant Secretary Fish and Wildlife and Parks, Department of the Interior

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Attachments:

List of References Mentioned in Text

Table 1: C14 Samples and Radiocarbon Dates from Kennewick Skeletal Remains

Attachment 1. Hood, Darden (1999a) Report of sample processing and dating. Letter to Dr. Francis P. McManamon, 17 October 1999.

Attachment 2. Hood, Darden (1999b) Additional information regarding Beta Analytic's radiocarbon dating analysis of Kennewick bone sample CENWW.97.R.24(Mta)/DOI1a. Letter to Dr. Francis P. McManamon, 18 November 1999.

Attachment 3. R. E. Taylor (1999) Results of the UCR Radiocarbon Analysis of two Kennewick Bones Compared with the Earlier Results. Fax to Dr. Francis P. McManamon, 20 December 1999.

Attachment 4. Donahue, Douglas (2000a) Carbon-isotope measurements on the Kennewick bone. Letter to Francis P. McManamon, 10 January 2000; and Donahue, Douglas (2000b) Results of measurements, equipment preparation, sampling procedure, and the pretreatment procedure. Letter to Francis P. McManamon, 9 January 2000.

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References Mentioned in Text

Donahue, Douglas (2000a) Results of the carbon-isotope measurements on the Kennewick. Letter to Francis P. McManamon, 10 January 2000. Included here as Attachment 4. On file, Archeology and Ethnography, National Park Service, Department of the Interior.

Donahue, Douglas (2000b) Results of measurements to date on the Kennewick bone, equipment preparation, sampling procedure, and the pretreatment procedure. Letter, 9 January 2000. Included here as Attachment 4. On file, Archeology and Ethnography, National Park Service, Department of the Interior.

Egan, James E. (2000) Letter to Allison B. Rumsey, Department of Justice, January 10, 2000.

Fagan, John L. (1999) Analysis of Lithic Artifact Embedded in the Columbia Park Remains. Report at http://www.cr.nps.gov/aad/kennewick/fagan.htm, visited December 8, 1999. Archeology and Ethnography Program, National Park Service, Department of the Interior.

Hood, Darden (1999a) Report of sample processing and dating. Letter to Dr. Francis P. McManamon, 17 October 1999. Included here as Attachment 1. On file, Archeology and Ethnography Program, National Park Service, Department of the Interior.

Hood, Darden (1999b) Additional information regarding Beta Analytic's radiocarbon dating analysis of Kennewick bone sample CENWW.97.R.24(Mta)/DOI1a. Letter to Dr. Francis P. McManamon, 18 November 1999. Included here as Attachment 2. On file, Archeology and Ethnography Program, National Park Service, Departmen: of the Interior.

Hood, Darden (1999c) Background about the Kennewick C14 Samples. E-Mail to Dr. Francis P. McManamon, 9 December 1999. Included here as Attachment. On file, Archeology and Ethnography Program, National Park Service, Department of the Interior.

Huckleberry, Gary, Thomas W. Stafford, and James C. Chatters (1998) Preliminary Geoarchaeological Studies at Columbia Park, Kennewick, Washington, USA. Report Submitted to U.S. Army Corps of Engineers, Walla Walla District, March 23, 1998.

Huckleberry, Gary and Julie K. Stein (1999) Analysis of Sediments Associated with Human Remains Found at Columbia Park, Kennewick, WA. Report at http://www.cr.nps.gov/aad/kennewick/huck_stein.htm, visited December 8, 1999. Archeology and Ethnography Program, National Park Service, Department of the Interior.

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McManamon, Francis P. (1999) The Initial Scientific Examination, Description, and Analysis of the Kennewick Man Human Remains. Report at <http://www.cr.nps.gov/aad/kennewick/mcmanamon.htm>, visited December 8, 1999. Archeology and Ethnography Program, National Park Service, Department of the Interior.

Powell, Joseph F. and Jerome C. Rose (1999) Report on the Osteological Assessment of the "Kennewick Man" Skeleton (CENWW.97.Kennewick). Report at <http://www.cr.nps.gov/aad/kennewick/powell_rose.htm>, visited December 8, 1999. Archeology and Ethnography Program, National Park Service, Department of the Interior.

R. E. Taylor (1999) Results of the UCR Radiocarbon Analysis of two Kennewick Bones Compared with the Earlier Results. Fax to Dr. Francis P. McManamon, 20 December 1999. Included here as Attachment 3. On file, Archeology and Ethnography Program, National Park Service, Department of the Interior.

R. E. Taylor, D. L. Kirner, J. R. Southon, and J. C. Chatters (1998) Letter to <u>Science</u>, "Radiocarbon Dates of Kennewick Man." <u>Science</u> 280:1171-1172.

Wakeley, Lillian D., William L. Murphy, Joseph B. Dunbar, Andrew G. Warne, Frederick L. Briuer, and Paul R. Nickens (1998) Geologic, Geoarchaeologic, and Historical Investigation of the Discovery Site of Ancient Remains in Columbia Park, Kennewick, Washington. Prepared for U.S. Army Engineer District, Walla Walla, WA (Technical Report GL-98-13), September 1998.

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ATTACHMENT 1

RADIOCARBON DATING SERVICES

Mr. DARDEN G. HOOD Director

October 17, 1999

RONALD E. HATFIELD Laboratory Manager

CHRISTOPHER PATRICK TERESA A. ZILKO-MILLER Associate Managers

Dr. Francis P. McManamon Dept. of Interior National Park Service Archeology And Ethnography Program 1849 C Street N.W. (NC 340/2275) Washington, DC 20240

Dear Dr. McManamon:

Please find enclosed the radiocarbon dating result for one bone sample "CENWW.97.R.24(MTa)/DOI1a" which was received on September 10. It was very small, requiring us to convert the sample carbon to graphite and then to count the radiocarbon atomically using an accelerator mass spectrometer (AMS). It provided plenty of carbon for reliable measurements and all analytical steps went normally. The quoted errors represent 1 sigma statistics. Since these errors cannot include uncertainties outside of those which can be quantified during measurement, it is best to consider them as minimum quotes.

Note that we notified your office upon beginning the analysis with an observation that the "R" in the submitter number on the sample package was not listed on the sample datasheet. Since it was listed on the sample package, we have used it in the reported sample designation number.

The bone sample was highly encrusted and in-filled with non-calcareous minerals. These minerals were physically eliminated with grinding, prior to demineralization of the apatite fraction with hydrochloric acid. The resultant protein extracted was subjected to alka i in high enough concentration to eliminate any secondary organic acid contamination. SEM analysis (photo-micrographs enclosed) were examined prior to pretreatment and after pretreatment (but prior to AMS analysis) to establish the integrity of the sample material.

The report sheet contains calibration results which enhance the accuracy of the radiocarbon dating. A hard-copy is enclosed showing the radiocarbon year/calendar year correlation curve segment associated with the radiocarbon date, along with explanation sheets. You will notice the X axis (cal BC age) that multiple two sigma ranges are possible for the radiocarbon date. This is discussed on the report sheet.

The results are reported in three formats; the Conventional Radiocarbon Age (BP) which is systematic with radiocarbon dates quoted without calendar calibration, calibrated calendar age (cal BC) which is corrected for true half life and atmospheric fluctuations and reported in calendar years, and calibrated Conventional Radiocarbon Age (cal BP), where the same half life and atmospheric fluctuation corrections are applied to provide a corrected BP format result (BP = before present, present being AD 1950). The cal BC and cal BP results are reported using the

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two sigma, 95% probability limitation. As noted on the report sheet, if other lines of evidence give you confidence to use the one sigma range on the calibrated results, you may use that range instead (which is listed on the hard-copy calibration print-out). In summary, the results are:

Conventional Radiocarbon Age:	8410 +/- 40 BP
Calibrated Calendar Age (2 sigma):	cal BC 7560 to 7455 and cal BC 7395 to 7370
Calibration Radiocarbon Age (2 sigma):	cal BP 9510 to 9405 and cal BP 9345 to 9320

Also enclosed is a Quality Assurance report showing the expected and measured ages for standards and a blind measured in the AMS. As I previously mentioned, we only rely on the AMS for the measurement. The machine is provided with our own standards, blanks, and blinds, already loaded in the target holder. The machine simply makes a measurement for us, which we verify. The QA report shows the measurement of two secondary standards (TIRI wood and TIRI turbidite). These two targets are international standards, with known consensus values. The "expected values" listed on the report are those consensus values. The "blind" listed on the QA report is a sample which had been previously analyzed by us. The AMS facility did not know the previous result for this blind.

A photo-documentary of the analysis is enclosed. Given the sensitivity of this analysis, each step of the analysis was carefully documented. Notes were taken by each individual involved in the analysis which consisted of myself Mr. Darden Hood, Director (20 years experience), Mr. Ronald Hatfield, Laboratory Manager (18 years experience), Mr. Christopher Patrick, Associate Manager (15 years experience), Ms. Teresa Zilko-Miller (12 years experience), Ms. Lethia Cerda, Office Coordinator (8 years experience), and Mr. David Miller, Staff (6 years experience). The sample graphite along with the necessary standards, already pressed into the target holder under our control, was sent to the AMS facility at Lawrence Livermore National Laboratory for measurement, and the result verified through our QA program.

One comment on the results is the 13C/12C ratio result. The value is elevated, indicating the individual had a C4 plant, or marine diet. Corn is the staple diet of most individuals with an elevated 13C/12C ratio. Since corn was not present 9000 years ago (to our knowledge), it suggests the likelihood of a marine diet. If this is the case, the presence of a "reservor effect" in the diet may need to be considered. This effect may make the radiocarbon dating "too old" by some amount, perhaps by several hundred years.

The cost of the analysis was charged to your MASTERCARD. A receipt is enclosed. Also enclosed is excess poor quality bone which was not used in the analysis and the remaining protein extracted from the sample. As always, if you have any questions or would like to discuss the results, don't hesitate to contact me.

Darden Hood

Page 2 ATTACHMENT



DR. M.A. TAMERS and MR. D.G. HOOD

UNIVERSITY BRANCH 4985 S.W. 74 COURT MIAMI, FLORIDA, USA 33155 PH: 305/667-5167 FAX: 305/663-0964 E-MAIL: beta@radiocarbon.com

REPORT OF RADIOCARBON DATING ANALYSES

 Dr. Francis P. McManamon
 Report Date:
 October 17, 1999

 Department of Interior
 Material Received:
 September 10, 1999

 Sample Data
 ¹³C / ¹²C
 Conventional Ratio

 Beta-133993
 -12.6 o/oo
 8410 +/- 40 BP

 SAMPLE =: CENWW.97.R.24(MTa)/DOIIa ANALYSIS: Standard-AMS MATERIAL/PRETREATMENT:(bone collagen): collagen extraction with alkali

COMMENT:

The above noted Conventional Radiocarbon Age can be calibrated to enhance the accuracy of the result. Our calendar calibrations are now calculated back to about 19,000 years using the newest calibration data as published in *Radiocarbon*, Vol. 40, No. 3, 1998 using the cubic spline fit mathematics as published by Talma and Vogel, *Radiocarbon*, Vol. 35, No. 2, pg 317-322, 1993: A Simplified Approach to Calibrating C14 Dates. Results are reported both as cal BC and cal BP. It is important to quote the original Conventional Radiocarbon Age, 13C/12C ratio and the calibration references in your publications for future reference by other researchers.

The equivalent calibrated calendar age (using the two sigma, 95% probability) in years BC is;

"cal BC 7560 to 7455 and cal BC 7395 to 7370"

The equivalent calibrated radiocarbon age (using the two sigma, 95% probability) in years BP is;

"cal BP 9510 to 9405 and cal BP 9345 to 9320"

Two ranges are possible due to "wiggles" in the calibration curve in this time region. A graphical representation of this calibration is enclosed. The two sigma range is quoted to encompass the delineation between separate radiocarbon events. One sigma ranges may be more appropriate for your research if other lines of evidence allow the use of higher precision. The one sigma ranges are "cal BC 7535 to 7480 and cal BP 9485 to 9430".

These calibration results are unique to the single Conventional Radiocarbon Age. Multiple measurements of the sample would provide statistically indistinguishable radiocarbon ages, each with its own unique calibrated range. For this reason, it is recommended that the calibration results be used in general terms.

When comparing the statistical agreement between radiocarbon dates, it is best to compare Conventional Radiocarbon Ages, as the calibration results may vary depending on the calculation format and time of calibration (ie calibration tables have changed through the years). The best average for muliple dates is to calculate a weighted average for Conventional Radiocarbon Ages and then do the calibration.

DOI 05827

Dates are reported as RCYBP (radiocarbon years before present, "present" = 1950A.D.). By International convention, the modern reference standard was 95% of the C14 content of the National Bureau of Standards' Oxalic Acid & calculated using the Libby C14 half life (5558 years). Quoted errors represent 1 standard deviation statistics (68% probability) & are based on combined measurements of the sample, background, and modern reference standards.

Measured C13/C12 ratios were calculated relative to the PDB-1 international standard and the RCYBP ages were normalized to -25 per mil. If the ratio and age are accompanied by an (*), then the C13/C12 value was estimated, based on values typical of the material type. The quoted results are NOT calibrated to calendar years. Calibration to calendar years should be calculated using the Conventional C14 age.

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RADIOCARBON DATING SERVICES

Mr. DARDEN G. HOOD

RONALD E. HATFIELD Laboratory Manager

CHRISTOPHER PATRICK TERESA A. ZILKO-MILLER <u>Associate Managers</u>

Quality Assurance Report

This report provides the results of reference materials used to validate AMS radiocarbon dating results on unknown materials, prior to reporting. Unknowns and reference materials were chemically converted to graphite at Beta and then sent to CAMS for C14 content measurement.

Reference standard results for Beta-133993		
Report date:	October 17, 1999	
Submitter:	Dr. Francis McManamon	
CAMS report:	October 4, 1999	

Secondary oxalic acid reference standard.

Expected value:	103.9 % modern
Measured value:	103.9 % +/- 0.3%
Agreement:	good

TIRI wood standard (international standard)

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Expected value:	4503 +/- "6" BP
vleasured value:	4510 +/- 30 BP
Agreement:	good

TIRI carbonate standard (international standard)

Expected value:	18,155 +/- "34" BP
Measured value:	18,390 +/- 70 BP
Agreement:	good

Blind sample (measured radiometrically at Beta Analytic and sent to CAMS without their knowledge of the previous result).

Radiometric age at Beta: AMS age at CAMS: Agreement: 1160 +/- 60 BP 1150 +/- 40 BP good

Background material:

(double-spar calcite) Expected value: greater than 50,000 BP Measured value: 56500 +/- 600 BP Agreement: good

(Miocene Coal) Expected value: 50,000 BP Measured value: 47000 +/-270 BP Agreement: good

Validation:

DOI 05828

4985 S.W. 74 COURT, MIAMI, FL 33155 U.S.A. TELEPHONE: 305-667-5167 / FAX: 305-663-0964 / INTERNET: beta@radiocarbon.com WEB SITE: http://www.radiocarbon.com

Date And

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CALIBRATION OF RADIOCARBON AGE TO CALENDAR YEARS





Beta Analytic Radiocarbon Dating Laboratory

4985 S.W. 74th Court, Miami, Florida 33155 • Tel: (305)667-5167 • Fax: (305)663-0964 • E-mail: beta@radiocarbon.com

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RADIOCARBON DATING SERVICES

Mr. DARDEN G. HOOD <u>Director</u>

RONALD E. HATFIELD Laboratory Manager

ANALYTICAL PROCEDURES AND FINAL REPORT

ل مجتهر المحتات

CHRISTOPHER PATRICK TERESA A. ZILKO-MILLER Associate Managers

FINAL REPORT

This package includes the final date report, this statement outlining our analytical procedures, a glossary of pretreatment terms, calendar calibration information, billing documents (containing balance/credit information and the number of samples submitted within the yearly discount period), and peripheral items to use with future submittals. The final report includes the individual analysis method, the delivery basis, the material type and the individual pretreatments applied. Please recall any correspondences or communications we may have had regarding sample integrity, size, special considerations or conversions from one analytical technique to another (e.g. radiometric to AMS). The final report has also been sent by fax or e-mail, where available.

PRETREATMENT

Results were obtained on the portion of suitable carbon remaining after any necessary chemical and mechanical pretreatments of the submitted material. Pretreatments were applied, where necessary, to isolate ¹⁴C which may best represent the time event of interest. Individual pretreatments are listed on the report next to each result and are defined in the enclosed glossary. When interpreting the results, it is important to consider the pretreatments. Some samples cannot be fully pretreated making their ¹⁴C ages more subjective than samples which can be fully pretreated. Some materials receive no pretreatments. Please read the pretreatment glossary.

ANALYSIS

Materials measured by the radiometric technique were analyzed by synthesizing sample carbon to benzene (92% C), measuring for ¹⁴C content in a scintillation spectrometer, and then calculating for radiocarbon age. If the Extended Counting Service was used, the ¹⁴C content was measured for a greatly extended period of time. AMS results were derived from reduction of sample carbon to graphite (100 %C), along with standards and backgrounds. The graphite was then sent for 14 C measurement in an accelerator-mass-spectrometer located at one of six collaborating research facilities, who return the results to us for verification, isotopic fractionation correction, calendar calibration, and reporting.

THE RADIOCARBON AGE AND CALENDAR CALIBRATION

The "Conventional C14 Age (*)" is the result after applying C13/C12 corrections to the measured age and is the most appropriate radiocarbon age (the "*" is discussed at the bottom of the final report). Applicable calendar calibrations are included for organic materials and fresh water carbonates between 0 and 10,000 BP and for marine carbonates between 0 and 8,300 BP. If certain calibrations are not included with this report, the results were either too young, too old, or inappropriate for calibration. DOI 05830

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WEB SITE: http://www.radiocarbon.com

PRETREATMENT GLOSSARY

Pretreatment of submitted materials is required to eliminate secondary carbon components. These components, if not eliminated, could result in a radiocarbon date which is too young or too old. Pretreatment does not ensure that the radiocarbon date will represent the time event of interest. This is determined by the sample integrity. The old wood effect, burned intrusive roots, bioturbation, secondary deposition, secondary biogenic activity incorporating recent carbon (bacteria) and the analysis of multiple components of differing age are just some examples of potential problems. The pretreatment philosophy is to reduce the sample to a single component, where possible, to minimize the added subjectivity associated with these types of problems.

"acid/alkali/acid"

The sample was first gently crushed/dispersed in deionized water. It was then given hot HCI acid washes to eliminate carbonates and alkali washes (NaOH) to remove secondary organic acids. The alkali washes were followed by a final acid rinse to neutralize the solution prior to drying. Chemical concentrations, temperatures, exposure times, and number of repetitions, were applied accordingly with the uniqueness of the sample. Each chemical solution was neutralized prior to application of the next. During these serial rinses, mechanical contaminants such as associated sediments and rootlets were eliminated. This type of pretreatment is considered a "full pretreatment". On occasion the report will list the pretreatment as "acid/alkali/acid - insolubles" to specify which fraction of the sample was analyzed. This is done on occasion with sediments (See "acid/alkali/acid - solubles"

Typically applied to: charcoal, wood, some peats, some sediments, textiles

"acid/alkali/acid - solubles"

On occasion the alkali soluble fraction will be analyzed. This is a special case where soil conditions imply that the soluble fraction will provide a more accurate date. It is also used on some occasions to verify the present/absence or degree of contamination present from secondary organic acids. The sample was first pretreated with acid to remove any carbonates and to weaken organic bonds. After the alkali washes (as discussed above) are used, the solution containing the alkali soluble fraction is isolated/filtered and combined with acid. The soluble fraction which precipitates is rinsed and dried prior to combustion.

"acid washes"

Surface area was increased as much a possible. Solid chunks were crushed, fibrous materials were shredded, and sediments were dispersed. Acid (HCI) was applied repeatedly to ensure the absence of carbonates. Chemical concentrations, temperatures, exposure times, and number of repetitions, were applied accordingly with the uniqueness of each sample. The sample, for a number of reasons, could not be subjected to alkali washes to ensure the absence of secondary organic acids. The most common reason is that the primary carbon is soluble in the alkali. Dating results reflect the total organic content of the analyzed material. Their accuracy depends on the researcher's ability to subjectively eliminate potential contaminants based on contextual facts.

Typically applied to: organic sediments, some peats, small wood or charcoal, special cases

"collagen extraction"

The material was first tested for friability ("softness"). Very soft bone material is an indication of the potential absence of the collagen fraction (basal bone protein acting as a "reinforcing agent" within the crystalline apatite structure). It was then washed in de-ionized water and gently crushed. Dilute, cold HCI acid was repeatedly applied and replenished until the mineral fraction (bone apatite) was eliminated. The collagen was then dissected and inspected for rootlets. Any rootlets present were also removed when replenishing the acid solutions. Where possible, usually dependent on the amount of collagen available, alkali (NaOH) was also applied to ensure the absence of secondary organic acids.

Typically applied to: bones

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BETA ANALYTIC INC. RADIOCARBON DATING LABORATORY CALIBRATED C-14 DATING RESULTS

Calibrations of radiocarbon age determinations are applied to convert BP results to calendar years. The short term difference between the two is caused by fluctuations in the heliomagnetic modulation of the galactic cosmic radiation and, recently, large scale burning of fossil fuels and nuclear devices testing. Geomagnetic variations are the probable cause of longer term differences.

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The parameters used for the corrections have been obtained through precise analyses of hundreds of samples taken from known-age tree rings of oak, sequoia, and fir up to about 10,000 BP. Calibration using tree-rings to about 12,000 BP is still being researched and provides somewhat less precise correlation. Beyond that, up to about 20,000 BP, correlation using a modeled curve determined from U/Th measurements on corals is used. This data is still highly subjective. Calibrations are provided up to about 19,000 years BP using the most recent calibration data available (Radiocarbon, Vol 40, No. 3, 1998).

The Pretoria Calibration Procedure (Radiocarbon, Vol 35, No. 1, 1993, pg 317) program has been chosen for these calendar calibrations. It uses splines through the tree-ring data as calibration curves, which eliminates a large part of the statistical scatter of the actual data points. The spline calibration allows adjustment of the average curve by a quantified closeness-of-fit parameter to the measured data points. A single spline is used for the precise correlation data available back to 9900 BP for terrestrial samples and about 6900 BP for marine samples. Beyond that, splines are taken on the error limits of the correlation curve to account for the lack of precision in the data points.

In describing our calibration curves, the solid bars represent one sigma statistics (68% probability) and the hollow bars represent two sigma statistics (95% probability). Marine carbonate samples that have been corrected for δ 13/12C, have also been corrected for both global and local geographic reservoir effects (as published in Radiocarbon, Volume 35, Number 1, 1993) prior to the calibration. Marine carbonates that have not been corrected for δ 13/12C are adjusted by an assumed value of 0 ‰ in addition to the reservoir corrections. Reservoir corrections for fresh water carbonates are usually unknown and are generally not accounted for in those calibrations. In the absence of measured δ 13/12C ratios, a typical value of -5 ‰ is assumed for freshwater carbonates.

(Caveat: the correlation curve for organic materials assume that the material dated was living for exactly ten years (e.g. a collection of 10 individual tree rings taken from the outer portion of a tree that was cut down to produce the sample in the feature dated). For other materials, the maximum and minimum calibrated age ranges given by the computer program are uncertain. The possibility of an "old wood effect" must also be considered, as well as the potential inclusion of younger or older material in matrix samples. Since these factors are indeterminant error in most cases, these calendar calibration results should be used only for illustrative purposes. In the case of carbonates, reservoir correction is theoretical and the local variations are real, highly variable and dependant on provenience. Since imprecision in the correlation data beyond 10,00 years is high, calibrations in this range are likely to change in the future with refinement in the correlation curve. The age ranges and especially the intercept ages generated by the program, must be considered as approximations.) Page 8 ATTACHMENT



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Beta Analytic Radiocarbon Dating Laborator XTTACHMENT 4985 S.W. 74th Court, Miami, Florida 33155 + Tel: (305)867-5167 " Fax: (305)663-0964 + E-mail: beta & radio:arbon.com

RADIOCARBON DATING SERVICES

Dr. MURRY A. TAMERS Mr. DARDEN G. HOOD <u>Directors</u>

RONALD E. HATFIELD Laboratory Manager

CHRISTOPHER L PATRICK TERESA A. ZILKO-MILLER Associate Managers

SCANNING ELECTRON MICROSCOPY (SEM) of materials submitted for radiocarbon dating

Scanning Electron Microscopy (SEM) can be used to magnify objects up to 10,000 times. SEM photographs showing microscopic details provide very useful information in the interpretation of radiocarbon dates. For instance, SEM can be used to distinguish primary vs. secondary shell structure and to identify very small wood, charceal, and carbonate samples. SEM micrographs are also an excellent addition to reports and theses. We highly recommend this analysis through your own sources, or if not available, by our services.



Primary CaCO3, 690x, SEM



Secondary CaCO3, 690x, SEM



Samples and pencil point 6x, light photo



Cedar or cypress, 180x, SEM



Planktonic foraminifera, 95x, SEM



CaCO3 foram infilling, 1360x, SEM

<u>APPROPRIATE MATERIALS</u>: SEM is especially useful for AMS samples. It is recommended for: (1) very small carbonates which cannot be pretreated (forams, ostracods, coccoliths); (2) unidentified macro-fossils concentrated from sediments; and (3) wood or charcoal for which some taxon identification is useful.

THE SERVICE & COST: Three (3) micrographs of various angles and/or magnifications are provided for each sample. Micrographs are obtained on a representative portion of the material submitted for radiocarbon dating, not on the dated material itself. The technician will usually be able to choose the angles and magnifications which are most appropriate. The service does not include identification or characterization, but wherever possible, some will be provided.

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Beta Analytic Inc.

SEM photo micro-graphs prior to chemical pretreatments.



23. Freshly broken bone surface.



24. Crystalline bone structure in un-pretreated sample.



25. Sediment in-filling, prior to removal (non-calcareous) with visible primary bone material.

Four views of the pretreated protein extract. No calcium or phosphorus phase remaining (ie the apatite was effectively removed) and no visible organic contaminants were observed. Only secondary component observed was a trace amount of alumino-silicate minerals, possibly K-feldspar.



26.





29.



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Consistent Accuracy Delivered On Time. Beta Analytic Inc. 4985 SW 74 Court Miami, Florida 33155 USA Tel: 305 667 5167 Fax: 305 663 0964 beta@radiocarbon.com www.radiocarbon.com

ATTACHMENT Z

DR. MURRY TAMERS MR. DARDEN HOOD Co-directors

1.

Mr. Ronald Hatfleid Laboratory Manager

Mr. Christopher Patrick Ms. Teresa Ziiko-Miller Assocate Managers

November 18, 1999

Dr. Francis P. McManamon Dept. of Interior National Park Service Archaeology And Ethnography Program 1849 C Street N.W. (NC 340/2275) Washington, DC 20240

Dear Dr. McManamon:

We received a telephone call from Jason Roberts requesting additional information regarding our radiocarbon dating analysis of your bone sample " CENWW.97.R.24 (Mta)/DOI1a".

The questions were:

1. What was the collagen content of the originally submitted bone?

The original weight of the bone was 9.1 grams. The amount of collagen extracted was 0.030 grams (30.0 mg). The relates to a percent concentration of 0.3%. The value is very low due to the high mineral content of the submitted bone.

2. What was the carbon concentration within the extracted collagen?

9.5 mg of the collagen was used for the analysis. This provided us with 3.2 mg of carbon. The percentage carbon is then calculated as 33.7% carbon within the collagen.

If I can answer any further questions, please do not hesitate to contact me.

Sincerely,

Lew Horo

Darden Hood Director

ATTACHMENT 2

FECH : P.E. TAYLOR

PHONE NO. : 909 799 3703

ATTACHMENT 3

FAX MESSAGE

R. E. Taylor Radiocarbon Laboratory / Archaeometry Laboratory Department of Anthropology University of California, Riverside, CA 92521 (909) 787-5521 / FAX (909) 787-5409 retaylor@citrus.ucr.edu

DATE: December 20, 1999

TO: Dr. Frank McManamon

FAX: (202) 343-5260

RE: (1) UCR Kennewick results (2) responses to your inquiries of 12/7/99 and 12/17/99

Pages Transmitted: 6 pages + table = 7 pages

Dear Frank:

Attached as a table are the results of the UCR ¹⁴C analysis of two Kennewick bones compared with our earlier Kennewick results for comparison.

1. Comments on the UCR ¹⁴C Results: On the basis of their amino acid carbon contents (AACC) and amino acid profiles, UCR-3806 and 3807 exhibit much lower collagen (protein) preservation than the earlier Kennewick bone my lab previously analyzed (UCR-3476). UCR-3806 has totally lost its collegen-like amino acid pattern. As I reported previously, both UCR-3806 and UCR-3807 exhibited unusual amounts of effervescence in acid which is usually an indication of significant amounts of secondary carbonates and there was unusual difficulty in filtering the hydrolysates.

The AACC that I reported earlier by email has been revised in light of additional analyses. (As I mentioned to you previously, we had just received our new HPLC and were still calibrating with standards when the initial analyses were obtained.) The revised AACC values do not change the fact that both bones are problematical in terms of their suitability to yield accurate bone ¹⁴C values due to their degraded biogeochemical condition. Although UCR-3807 turns out to have more protein that I reported earlier (14.3% AACC of our modern bone standard), the amino acid composition is marginal in terms of its collagen- or non-collagen like characteristics. On a routine basis, our criteria for an acceptable bone is at least 5% AACC and where the bone retains a clear collagen-like amino acid profile. On the basis of their amino acid profiles, both UCR-3806 and UCR-3807 are classified as non-collagen.

Because of their biochemically degraded condition, I report the results of the ¹⁴C measurements in terms of "fraction modern" with the apparent ¹⁴C age cited in footnotes. You will also note that the reported $\delta^{13}C$ values of these two samples are not typical of collagen amino acids. I would interpret that these values reflect primarily a dietary effect--namely that the individual (assuming that there is only one individual here represented) subsisted largely on a marine diet (e.g., fish). There also could be a fractionation factor involved due to the poor protein preservation. (In the case of UCR-3476, the first Kennewick bone we ran, we also observed a depressed $\delta^{13}C$ value and, making certain assumptions, we calculated a reservoir corrected age of 7880±160 BP.)

In summary, UCR-3807 exhibits an younger age offset of about 3% (about 280¹⁴C years) in comparison with UCR-3476 while UCR-3806 is very anomalous with respect to UCR-3476. One

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UCR Kennewick Results Page 2

interpretation is that the age offsets reflect varying percentages of more recent and/or modern contamination in both UCR-3806 and UCR-3607, with the percentage contribution of contamination increasing as a function of the decreasing residual collagen protein content. For UCR-3807, there is enough residual collagen so that the offset is limited to a few percent, while for UCR-3806, the very low AACC is reflected in the much more recent anomalous age.

- 2. Responses to Questions:
- A. Questions of December 7
- (1) First set:
- 1. Did any of you observe any structure or other characteristics of the extracted carbon that indicates it is deteriorated collagen rather than an intrusive element?

Without sequencing data, it would be difficult to establish definitively that the amino acids came only from collagen peptides. The observation that the age offset increases in inverse relationship to the collagen content in both UCR-3806 and UCR-3607 strongly suggests that there are exogenous amino acids in these samples. As you know, in bone, it is usually assumed that the older the inferred ¹⁴C age the more likely that this is closer to the actual age since typically noncarbonate contamination that has not been sufficiently removed generally renders samples "too young."

2. Did any of you observe any structure or other characteristics of the extracted carbon that indicates that it is from a source external to the bone sample?

The SEM images did reveal some microstructures that we could not identify and thus it is not possible to determine if they were organic in nature. It was difficult to filter the hydrolysate of both UCR-3806 an UCR-3807 which is rarely a problem with high collagen yield bone such as UCR-3476.

3. In your experience, is it invariable/common/rare/impossible for "old" intrusive carbon to contaminate a bone sample from a riverine, floodplain, or lower river terrace geomorphologic context?

It entirely depends on the characteristics of the humic and other soil organic compounds contained in the soil together with the nature of the ground water conditions over the time period that the bone has been exposed to the environment. Also, can it be assumed that the bone was always buried in the same soil profile? May it have been exposed and then reburied as some unknown period in the past?

4. Are there other structural, physical, chemical, or visual characteristics of the sample and extracted carbon that suggest to you that it is uncontaminated?

On the contrary, the chemical state of the amino acid extract from UCR-3807, and especially that from UCR-3806, in my view, points strongly to the possibility that it may be contaminated with exogenous carbon compounds.

5. Are there other structural, physical, chemical, or visual characteristics of the sample and extracted carbon that suggest to you that it is contaminated? If so, what do you believe the contaminate is?

As noted in 4, the chemical state of the collagen in UCR-3807 and especially UCR-3806 raises

UCR Kennewick Results Page 3

> the strong possibility that both may be contaminated. Soil humics of various types are the most obvious candidates.

6. In your experience, what magnitude of time span would be required for the charac: eristics you observed in the extracted carbon from these samples to have deteriorated from normal bone collagen?

This is very difficult to determine since there are many environmental variables that can influence rates of biogeochemical diagenesis processes in bone structures.

7. Before we took the samples from the Kennewick remains in September, we consulted with experts, including each of you about the kind of bone to select. Dense bone in weight bearing areas and mid-shaft were the main suggestions we got and followed. If we were to take additional samples, is there a way to determine visually which bones would be rich in collagen? If not visually, what other means would be needed to detect collagen levels?

Except with highly degraded bone where there is a "chalk-like" appearance, it is usually difficult to determine which bones have retained more unaltered collagen on the basis of gross visual appearance. Some have used responses to ultraviolet to gauge collagen content but there are a number of variables that interfere with good responses. (I believe that I suggested previously to you that it would be very helpful to take very small amounts of bone from 20 different Kennewick bones and determine their amino acid composition. This would give you an objective basis on which to gauge differential preservation.)

- (2) Second Set
- 1. In your experience is it common or rare for samples from the same skeleton to display such a range in collagen structure and content?

Few specific experiments have addressed this directly. The Haverty skeletons exhibited significant variability in protein content but, in this case, the analyses was done on different skeletons that was assumed to have been buried in close spacial and temporal proximity. (Brooks, S., R. H. Brooks, J. Austin, G. Kennedy, J. R. Firby, L. A. Payen, C. A. Prior, P. J. Slota, Jr., and R. E. Taylor. 1991. The Haverty Human Skeletons: Morphologial, Depositional and Geochronological Characteristics. Journal of California and Great Basin Anthropology 12:60-83.). In cases where different parts of a skeleton have been subjected to different alternating ground water/moisture cycle (wet/dry/wet) regimes, there can be significant differences among the bones. This can occur if different parts of a skeleton are not being exposed to the same the ground water conditions or has been exposed to different soil types by redeposition.

2. Do you have any suggestions that could explain this difference reasonably?

As noted above, differential ground water cycle (wet/dry/wet) regimes could explains the difference in the same skeleton. Conditions would depend on the relationship between the position of different bones in the skeleton with reference to the soil profile/ground water regime, i.e., if different bones were exposed to varying soil/ground water conditions.





PHONE NO. : 909 799 3783

UCR Kennewick Results Page 4

B. 12/17 Question Set

 Have you or some other expert ever summarized the characteristics of skeletal remains earlier than 7000 years BP that have been dated? We are checking articles and books on the subject, such as articles by Powell and Steele that review early skeletal evidence; "Brule Woman" article; "Arlington Springs Woman" info; Windover site burial population; Pyramid Lake and Spirit Cave mummies; other?

There is an extensive literature on the ¹⁴C dating of bone and the problems of dealing with collagen degraded bone extending back for several decades. For example, Taylor 1987: 53-61 reviews the research as of the mid-1980s and cites the earlier literature. Hedges and Law 1989 and Hedges and Van Klinken 1992 are excellent overviews and presents the experiences of the Oxford Laboratory. Stafford *et al.* 1988 and 1991 reports extensive and excellent studies carried out by him at the Carnegie Geophysical Laboratory and at the University of Arizona. Taylor 1982, 1987b, 1992, 1994 reports some of the work of my lab. Burkey *et al.* 1998 reports our work in attempting to deal with collagen-degraded bone.

All of these studies highlight the significant variability in the degree to which endogenous carboncontaining fractions in bone are retained and are, or are not, protected from contamination by a wide variety of physical and chemical diagenetic mechanisms. It is widely acknowledged that obtaining accurate ¹⁴C age estimates on bone requires attention to detail in sample preparation and an appreciation that each bone may present an unique chemical challenge if the isolation of a fraction that contains only autochthonous carbon atoms is to be consistently achieved.

It should be reiterated that the biochemical condition of bone reflects more directly the diagenetic conditions to which it is exposed--which can be highly variable--so that, in one environment, 7,000, 10,000, or 40,000 year old bones can retain close to 100% of their *in vivo* collagen, while in another environment, a 1,000 year old bone may have lost most of its collagen content.

References:

Hedges, R. E. M. and I. A. Law: 1989. The radiocarbon dating of bone. Applied Geochemistry 4:249-233.

Hedges, R. E. M. and Van Klinken, G. J. 1992. A review of current approaches in the pretreatment of bone for radiocarbon dating by AMS. *Radiocarbon* 34:279-291.

Stafford, T. W., Jr., K. Brendel, and R. C. Duhamel. 1988. Radiocarbon, ¹³C and ¹⁵N analysis of fossil bone: Removal of humates with XAD-2 resin. *Geochimica et Cosmochimica Acta* 52: 2197-2206.

Stafford, T. W., Jr. P. E. Hare, L. Currie, A. J. T. Jull and D. J. Donahue. 199. Accelerator radiocarbon dating at the molecular level. *Journal of Archaeological Sciences* 18:35-72.

Taylor, R. E. 1982. Problems in the radiocarbon dating of bone. In Nuclear and Chemical Dating Techniques. L. A. Currie, ed., pp. 453-473. Washington, D.C.: American Chemical Society.

Taylor, R. E. 1987a. Radiocarbon Dating: An Archaeological Perspective. New York: Academic Press.



UCR Kennewick Results Page 5

> Taylor, R. E. 1987b. AMS ¹⁴C Dating of critical bone samples: Proposed protocol and criteria for evaluation. Nuclear Instruments and Methods in Physics Research B29:159-153.

Taylor, R. E. 1992. Radiocarbon Dating of Bone: Beyond Collagen. In R. E. Taylor, A. Long. and R. Kra, eds. Radiocarbon After Four Decades: An Interdisciplinary Perspective, pp. 375-402. New York: Springer-Verlag.

Burky, R. R., D. L. Kirner, R. E. Taylor, P.E. Hare, and J. R. Southon. 1998. Radiocarbon Dating of Bone Using Gamma-Carboxyglutamic Acid and Alpha-Carboxyglycine (Aminomalonate). Radiocarbon 40:11-20.

2. For these relatively ancient remains (post 7000) is the collagen and its structure typically deteriorated? Is the amount of carbon in the bones that is available for CI4 dating consistently low, if not consistently low, what seems to be cause of the variation?

As noted previously, there are many environmental variables that can influence rates of biogeochemical diagenesis. In most cases, the most critical variables are probably effective mean annual temperature and effective moisture. Typically, bone in tropical contexts is rapidly biochemically and physically degraded. Bone from cold environments, e.g. arctic or high altitudes and bone from special environments that excludes water (e.g., La Brea Tar Pits or in desiccated desert caves or rock shelters) can retain their collagen content for extended periods of time measured, in some cases, in excess of several tens of thousands of years.

3. Can you point me to any general or summary statements in your articles or radiocarbon texts and general articles about bone carbon deterioration over time, any graphs or tables on this?

Please see the comments on question 1 above.

4. In the processing of the bone samples has your lab needed to use all the bone?. If so, is this because of the deterioration of the collagen carbon, if not what factor has required use of most of the bone?

We used about 20% of the UCR-3807 bone we received and about 30% of UCR-3806 to obtain our dates. (We will need most of the remaining bone to undertake the additional studies to determine the source of the contamination. Please see answer to the next question.)

5. Can you explain to me in writing the dating of additional fractions that you and I have discussed, what do we hope to learn from this, will it be done with both samples or only the most deteriorated? How long do you estimate it will take?

As we discussed, I would like to determine, if possible, where the contamination is coming from. The most likely candidate is the humic fraction. We wish to do an XAD-extraction and also look directly at a total humic fraction. It may be necessary to request additional bone to do these tests, but we will start on the remaining bone currently in the lab. This may take up to another month to 6 weeks, depending on the problems we encounter.

6. What description is available of the first Kennewick sample from the Benton Co. coroner? What portion of the bone remained after the sample extraction at UCR?

All we have by way of a description of the first Kennewick sample is the paperwork that we received from the submitter. Our results were published in Science. [Taylor, R. E. et al. (1998) Science 280:1171-1172].

DOI 05841

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FROM : F.E. TAYLO

PHONE NO. : 989 799 3783

Dec. 20-1999 01:47P

UCR Kennewick Results Page 6

I trust these responses and suggestions have been responsive and helpful. If and when this data is released to the popular press, I know that you will find some way to get them to report it appropriately.

Regards,

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TAYLOR

Laboratory Sample Number Designation	Sample	Bone Preservation ⁴	Fraction measured	δ ¹³ C (permil)	Radiocarbon analysis	
	Designation				F.b	"C age (BP)
UCR-3476/ CAMS-29578	Sth left metacarpal APS-CPS-01	68.8%(C)	total amino acids	-15.4		8410±60 ⁴
UCR-3807/ CAMS-60684	CENWW.97.R.24/Mta)	14.3%(NC) ⁴	total amino acids	-10.8	0,3633±0.0014	^
UCR-3806/ CAMS-60683	CENWW.97.L.20b-DOI2t	2.3%(NC) ^r	total amino acids	-10.3	0.4216±0.0015	£

UCR/CAMS Radiocarbon Analyses of Kennewick Human Bone

"Expressed as % of amino acid carbon content (AACC) of modern bone standard. C = collagen-like amino acid composition NC = non-collagen amino acid composition

 ${}^{b}F_{m} = \text{fraction modern where } 1.0 = \text{"modern."} pM (percent modern) = F_{m} \times 100.$

'Conventional radiocarbon age in ¹⁴C years BP. Reservoir corrected age = 7880±160 [Taylor et al. (1998) Science 280:1171-1172] "Revised AACC after duplicate analysis and recalibration of HPLC. Initial analysis = 3.2% AACC of modern bone standard. Gly/Glu ratio and other indices of collagen-like amino acid profile indicates significant biogeochemical diagenesis has occurred and on this basis the profile is characterized as non-collagen.

*Apparent ¹⁴C age = 8130±40 BP

¹ Revised AACC after duplicate analysis and recalibration of HPLC. Initial analysis = 5.3% AACC of modern bone standard *Apparent ¹⁴C age = 6940±30 BP

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THE UNIVERSITY OF Tucson Arizona

NSF-Arizone AMS Facility العاقة عائلا فالعام المحافظ 1118 E. 4th St. Tucson, Arizona 85721-0081 Telephone: (520) 621-6810 Fussimile: (520) 621-0619 AMS@physics anzona.edu

ATTACHMENT 4

10 January 2000

Frank McManamon National Park Service 1849 C Street, NW Room NC-340 Washington, DC, 20248

Dear Dr. McManamon:

Attached are the results of carbon-isotope measurements on the Kennewick bone sample, which we have given the identification number AA34818, Sample B. The treatments of this sample are described in detail in my message to you of 13 December, 1999, and forwarded to you today. The sample from which the attached results were obtained is the one labeled "Sample B" in that message.

I am anxious to make several comments.

1.) The carbon yield for this sample was 0.05%. The yield is defined as the mass of carbon obtained after all of the treatments of the hone have completed, divided by the initial mass of bone used.

2.) This is well below the yield for which we would usually quote a result. In fact, for bones with a yield as low as this, we generally will not even make a radiocarbon measurement.

3.) Because of the unusual nature of this sample, we have indeed made a radiocarbon measurement of the carbon obtained from it, and the result of that measurement is on the attached report.

4.) I emphasize that, because of the low yield, we do not have confidence in the result. Since contamination would most probably be more recent than the bone material, we would expect that our result is a limit, and represents a minimum of the radiocarbon age.

We are certainly very interested in measurements on the Kennewick bone. Please keep us posted, and if further measurements are to be made, we would be anxious to participate.

Sincerely.

Nong Nonahme

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DOI 05844

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http://www.physics.arizona.edu/ams/index.html

Data Summary	
AMS Results: McManamon, F.	(Kinnewick Man)

10-Jan-00

SampleID δ13 FM 14C age (BP) Calibra_2 sigma AA# AA34818 -21.9 0.4889+-0.0066 5,750+-100BP 4800-4360BC wtd. avg.

Reported by: Nouglan Nonahac

Page 2 ATTACHMENT 4

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NSF-Anzona AMS Facility Physics Bldg, 81 1118 E. 4th St. Tucson, Arizona 85721-0081 Telephone: (520) 621-6810 Facsimile: (520) 621-9619 AMS@physics.artona.edu

9 January 2000

Frank McManamon National Park Service 1849 C Street, NW Room NC-340 Washington, DC, 20248

Dear Dr. McManamon:

Given below is a copy of the report concerning your Kennewick bone sample which I sent to you on 13 December, 1999. We have labeled the sample AA34818. Under separate cover is also a report of carbon-isotope measurements made on the sample.

Results of measurements to date on the Kennewick bone.

Equipment Preparation

The following equipment was used to perform various stages of sample preparation: 1) a Mettler H54AR scale; 2) a drying oven; 3) a Dremel tool; 4) aluminum foil; 5) cutting blade; 6) acetone; 7) distilled water; 8) autoclave; 9) two-ended stainless steel spatula; 10) stainless steel tweezers; 11) chem wipes; 12) VWR 4x4 weighing paper; 13) agate mortar and pestle; 14) glass scintillation vials; 15) 50 ml test tubes with lids; 16) Erlenmeyer filtration flask with rubber stopper; 17) water bath; 18) exacto knife; 19) stainless steel wood carving tools

The following were cleaned with acetone, rinsed with distilled water, and loaded into the drying oven for ~30 minutes: 1) aluminum foil; 2) spatula; 3) tweezers; 4) mortar and pestle; 5) exacto knife and new blade 6) wood carving tools.

After the tools had dried they were placed in a cleaned (acetone and DI-H2O rinsed) plastic tray with lid.

The following were cleaned in the autoclave: 1) spatula; 2) tweezers; 3) 50 ml test tube; 4) filtration flask.

After the implements were removed from the autoclave they were placed in a plastic tray with lid. After the glassware was removed it was sealed with aluminum foil and kept in ziplock bags until it was directly used.

Page 2 ATTACHMENT 4

Sampling Procedure

Dr. Tim Jull cut the submitted sample, labeled AA34818, and with initial mass = 6.2 grams, into 4 individual pieces for processing. Dr. Jull wore non-powdered latex gloves and safety glasses and used a cleaned Dremel tool with diamond blade to slice the sample into 4 sub-samples. These were each placed into individual glass vials labeled A, B, C, and D. Small fragments and powder remaining from the sawing were also saved and placed into a glass vial labeled E. The masses of the samples were: Sample A, 1.29g; Sample B, 1.27g; Sample C, 1.34g; Sample D, 1.79g; Sample E, 0.5g.

Of these 4 sub-samples, Jeanette O'Malley selected sample "A" with Dr. Donahue and Mitzi DeMartino watching. Ms. O'Malley also selected a portion of material from Sample E for nitrogen analysis.

From sample "E", Ms. O'Malley, wearing non-powdered latex gloves, picked out clean white flakes from scrap material on weighing paper, using cleaned tweezers. These flakes were weighed on the scale until a weight of 5.79mg of material was obtained. These flakes were then poured from the weighing paper into an agate mortar and crushed to fine powder. This powder was then placed on new weighing paper and had a total mass of 5.58mg. It was then poured into a clean glass vial with lid, labeled only with the AA number. This sample was then taken off site for nitrogen analysis at an independent, private lab, where it was determined that the sample contained 0.07% nitrogen. This is approximately a factor of ten below the nitrogen content of a bone for which we would expect to make a successful radiocarbon measurement.

From sample "A", Ms. OMalley, wearing non-powdered latex gloves and dust mask, selected the largest fragment. This piece had one surface area that had been directly exposed to the environment. Thus the opposite portion of the fragment, from the interior of the bone shaft, was used for sampling. Using an exacto knife, fine flakes and powder were scraped from this interior surface. A final total of 0.63 grams was extracted.

The Pretreatment Procedure

These 0.63 grams were placed in a covered test tube with 20ml of 0.25N HCl. There was a strong reaction of effervescence observed. The sample was then sonicated for 20 minutes, in 0.25N HCl, at room temperature. The solution was decanted and fresh DI water added. This rinsing process was repeated until a neutral pH was achieved. The sample appeared to be mostly fluffy powder, with a little gcl.

This hydrolyzed sample was then put in 20ml of 0.01N HCl in a 60 degree C waterbath overnight. The sample had little visible change the next day, so the sample was then placed in a 60 degree C sonicator bath for 2 hours. The result of this treatment was an opaque suspension.

The suspension was then filtered through fiberglass filter paper and the resulting solution was decanted into a 50ml beaker and frozen. This beaker, containing the frozen liquid, was then placed on a freeze-dry apparatus overnight. The resulting solid material was a white chalky granule residue that was a bit sticky, which is NOT characteristic of collagen and indicated that a poor result would be obtained from the radiocarbon measurement.

The sample was weighed, and had a mass of 21.8 milligrams. This material was then combusted in an oxygen atmosphere. The combusted sample yielded 0.42 milligrams of

Page 4 ATTACHMENT 4

carbon, or a 1.9 % combustion yield. This low combustion yield (the combustion yield from collagen should be 35-40%) indicates that the product of the freeze-dry step contained considerable non-carbonaceous mineral material.

To summarize, the overall yield,

Y = carbon yield/initial bone mass = 0.42 mg/0.63 grams = 0.07 percent.

The entire procedure was repeated with a second portion of sample A. This portion had an mitial mass of 0.38 grains, and the carbon extracted from this sample gave a yield, Y = 0.05 percent.

We can make a measurement of the radiocarbon content of either of these samples, but because of the very low yields, we are hesitant to do so. We are continuing to work with Sample B, and will keep you informed of our progress.

Now In Normme Douglas Donahue Professor of Physics

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