Projectile Points at 45LI224

Confederated Tribes of the Colville Reservation

History/Archaeology Program

Prepared by, Eric Gleason, Jacqueline Cheung, Brenda Covington

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FY17 PWR-1979-17-WA-05 (LARO) FY18 PWR-1979-18-WA-05 (LARO)

Funded by the Bonneville Power Administration and Bureau of Reclamation for the Federal Columbia River Power System Cultural Resource Program.

Contains Sensitive Information –

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Introduction

Site 45LI224 is situated within the Plateau Culture Area, in north central Washington State and includes a dense and discrete lithic scatter, exposed and reworked by Lake Roosevelt reservoir induced erosion. The discrete lithic scatter manifests as a lagged out gravel ridge in the drawdown zone and is where most artifacts have been observed and collected. Artifacts thus far collected from the surface of the site are documented in Table 1.

Table 1. All Surface Collected Artifacts from 45LI224, Categories and Counts Based on Repository Inventory.

Artifact	**Collection Date**						**Material Types**
	1996	**2015**	**2016**	**2017 Testing**	**2018 Testing**	**Total**	
Debitage				122	220	342	CCS, Mudstone, Quartzite, Granitic
Projectile Point Base	1	4		9	4	18	CCS
Biface		5	1	21	20	47	Basalt, Mudstone, CCS
Projectile Point		2		1		3	CCS, Andesite
Projectile Point Mid-Section					2	2	CCS
Core				14	3	17	Mudstone, CCS
Bifacial Retouched Flake				2	2	4	CCS
Utilized Flake				1	1	2	CCS
Unifacially Retouched Flake				5	3	8	CCS
Shell				1		1	Mussel Shell
Cobble Tool					2	2	Basalt, and Unknown
Hammerstone				1	1	2	Quartzite, Basalt
Scraper				1	1	2	CCS
Tabular Knife				1		1	Quartzite
Totals	1	11	1	179	259	451	

Archaeological and Historical Services were the first to collect an artifact from the site, a projectile point base, (initially documented as a biface fragment) in 1996 while conducting initial site inventory and documentation (Galm et. al 1996:5.4). During brief site revisits in 2015, CCT H/A crews observed and collected a complete Windust style projectile point (see Appendix A – Artifact photographs, Figure 5), as well as a larger lanceolate style projectile point or knife (Appendix A – Artifact photographs, Figure 6), and several other projectile point and biface fragments. In 2017, during the field testing project and a subsequent revisit, 179 artifacts were observed on the ground surface and collected. In 2018, another 259 artifacts were observed on the ground surface and collected.

Most of the surface exposed artifacts were recovered from the gravel ridge area of the drawdown zone. The gravel ridge area of the site is a particularly dynamic part of the drawdown zone environment. The top of the gravel ridge is approximately 10 to 15 feet below 1290 ft AMSL/full pool. As such it is exposed by nearly every seasonal drawdown and occasionally during low water levels in late summer. Exposure can span up to

6 or 7 months every year. When the gravel ridge is inundated, waves generated by wind and recreational boat use continue to rework exposed lag deposits and undercut the 1290 ft AMSL/full pool cutbank along the reservoir margin. Cyclic exposure and inundation cycles and associated erosion resulting in reworking of deposits begs the question of how much redistribution of artifacts within lag deposits has taken place, and, is it possible to determine if current artifact distribution reflects pre reservoir deposits and artifact distribution?

Given the dynamic reservoir environment, and the realm of adverse effects to which 45LI224 is subjected, erosion and artifact displacement is inevitable and obviously evident. However, artifacts collected from the gravel ridge all have sharp edges, show no obvious impact scars or evidence of dulling that could indicate extensive translocation, and some retain a calcium carbonate coating on what would have been their *in situ* lower surface. Given the frequency of site revisits and the aforementioned characteristics, it is likely these artifacts were exposed relatively recently and their morphology has suffered limited impact.

Discussion

Surface collection at 45LI224 started in 1996, and, to date, 451 artifacts have been collected, the majority of which are lithic debitage. However, 31 artifacts (7% of the surface collection) have been identified, either in the field or at the Repository, as complete or fragmentary projectile points with diagnostic elements. During a post field examination of artifact photographs, an additional six (6) artifacts were identified as possible diagnostic projectile point fragments, preforms, or knives. Combined, this brings the total number of surface collected artifacts with diagnostic elements to 37, approximately 8% of the total surface collection. Photographs of 36 of these projectile points and bifaces are grouped together in a single illustration, Figure 1, at slightly smaller than actual size. After grouping the photographs of the artifacts together in the single illustration, 24 of the 36 artifacts were determined to contain sufficient elements to assign a point type or style designation.

The following discussion of the projectile points recovered to date from 45LI224 is a preliminary review and compilation of existing data. As recommended in the testing report (Gleason et al. 2020) and explained in the discussion below, in order to understand the lithic assemblage at 45LI224 relative to similar sites and assemblages in the region, a complete and rigorous lithic analysis is critical.

By looking at scaled photographs of these 36 artifacts (Figure 1) it becomes apparent that this collection is dominated by stemmed projectile points and projectile point fragments. The two mostly complete projectile points in this collection, artifacts 2015-001 and 2015-006, both have contracting stems and slight to nonexistent shoulders. Most of the fragments (n=27) appear to be basal fragments, although it is sometimes difficult to ascertain the orientation of a fragment based solely on a photograph. Of the basal fragments, 15[1] are limited to the stem or basal section, being either broken below the shoulder of the point, or from points with a very weak or nonexistent shoulder. The other 12 basal fragments[2] retain evidence of a shoulder. The two midsection fragments shown in Figure 1 (2015-11, 2018-157) have indistinct shoulders. Another projectile point midsection in the collection, 2018-215 (Appendix A – Artifact photographs, Figure 7), has no shoulders and is not diagnostic, and thus is not shown in Figure 1. The three complete leaf shaped bifaces (2017-013, 2017-213, 2018-153) in the collection could be projectile points, knives, or preforms. Two fragments also appear to be from similar sized bifaces (2017-051, 2018-036).

[1] Basal fragments without shoulders: 2017-009, 2017-119, 2018-089, 2018-121, 2017-214, 2015-009, 2017-031, 2017-025, 2017-060, 2017-085, 2018-057, 2017-175, 2018-197, 1996-001, 2016-001.

[2] Basal fragments with shoulders: 2017-116, 2018-059, 2015-010, 2018-205, 2018-206, 2017-080, 2015-004, 2015-007, 2015-008, 2018-188, 2018-082, 2015-002.

Figure 1. Grouped illustration of 45LI224 artifacts identified as diagnostic projectile points, projectile point fragments, possible projectile fragments, and bifacial preforms or knives.

The diagnostic lithic assemblage and style of most of the projectile points is consistent with the Windust Phase of the Columbia Plateau as defined in 1970 by Frank C. Leonhardy and David G. Rice and refined by Rice in 1972. In *The Windust Phase in Lower Snake River Region Prehistory,* Rice describes and compares the assemblages recovered from three early archaeological sites in the Lower Snake River Region: Windust Cave (45FR46), Marmes Rockshelter (45FR50), and Granite Point (45WT41). The locations of these sites in relation to 45LI224 and to several other Windust age sites is depicted in Figure 2. Rice looked at an assemblage of 1,328 artifacts recovered from the lowest deposits of the three sites. No dates were available for the collection from Windust Cave, however the stylistically contemporaneous components of the two other sites were radiocarbon dated to 10,000–8,000 BP (11,500–8,900 cal BP). Rice looked at 229 projectile points (136 complete and 93 basal fragments) from these sites, and divided them into 24 separate groups based on their morphology; examining traits such as the presence or absence of shoulders, and the shape of the blade, stem, and base. He also examined the flaking pattern, cross section, and material type (1972:36). E. S. Lohse (1985, 1994) condensed Rice's 24 groups into three subtypes, which he designated as Windust A, B, and C (Figure 3).

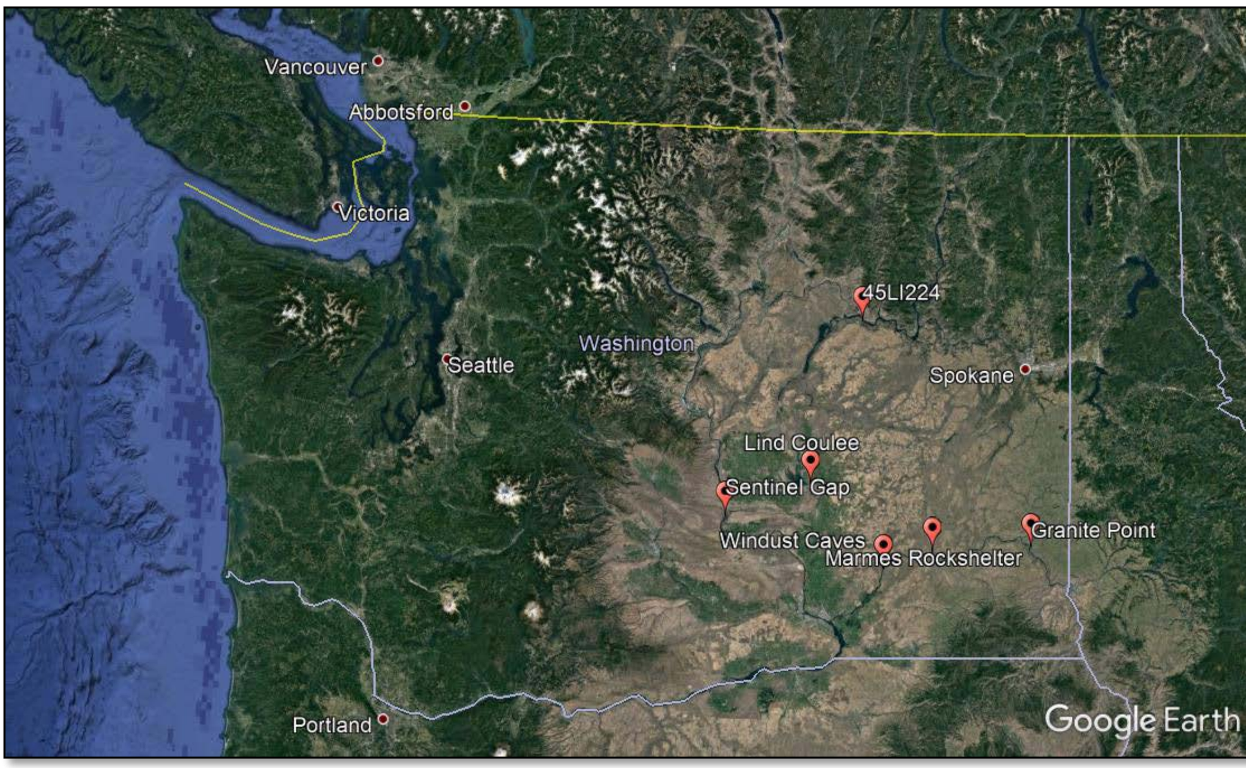

Figure 2. Map of Washington State showing Western Stem Tradition sites mentioned in the text.

Both Rice and Lohse recognized that Windust was not the sole stemmed point in the early assemblages of the West, and more specifically of the Columbia Plateau. Together these points have been more broadly referred to as part of the Western Stemmed Tradition (WST). E. S. Lohse and Coral Moser (2014) identified five distinct point styles in the WST of the Columbia Plateau, these are the three variations of Windust (A, B and C), together with points first identified at two other type sites, Lind Coulee (45GR97) in Washington's channeled scablands, and Haskett (10PR37) in eastern Idaho. These point types and their identifying morphological traits described by Lohse and Moser as elements of the WST, are used in this report to assign point types to the 45LI224 collection. Types identified as Windust A points are shouldered with a straight to slightly contracting stem and a straight base. Windust B points are also shouldered with a straight or slightly contracting stem, however they have a concave base. Windust C points do not have a shoulder, instead having a smooth transition from the blade to a contracting stem with a markedly concave base. Lind Coulee points consist of "…stemmed forms and shouldered forms with straight to contracting basal margins, and convex bases", while Haskett points are "…large lanceolate points with long contracting stems and slightly convex bases" (Lohse and Moser 2014:52).

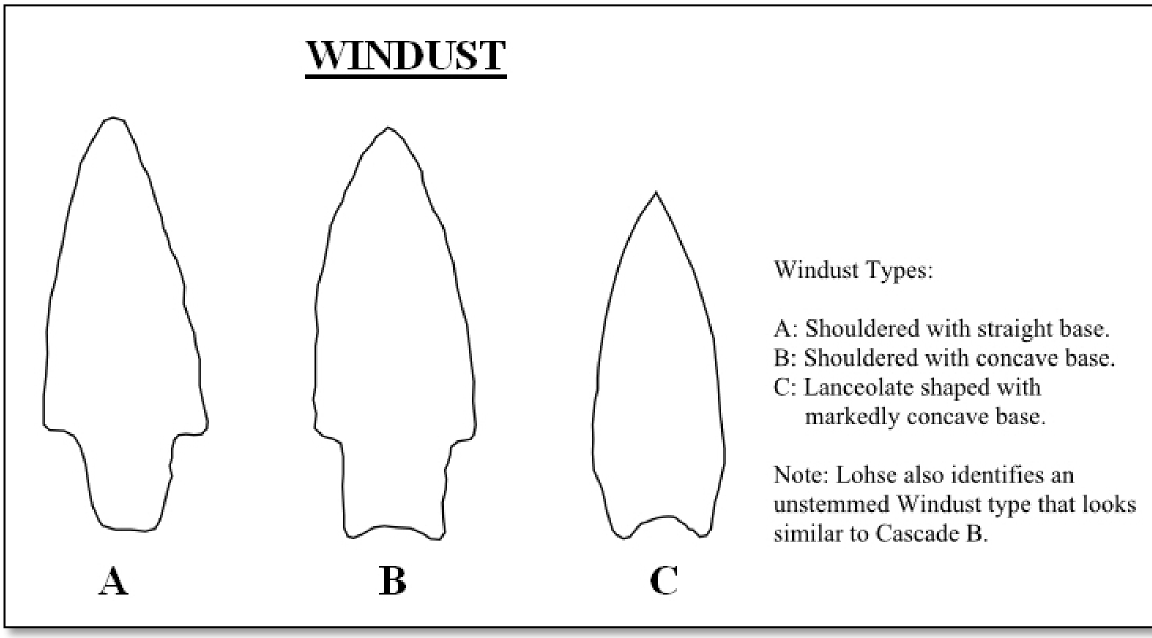

Figure 3. Stylized outlines of the three Windust subtypes based on Lohse (1985, 1994).

The WST began on the Columbia Plateau by at least 13,000 cal BP and extended to 11,000 cal BP, with a terminal date that is traditionally extended to 9,000 cal BP (Brown et al 2019:488-489). David Rice (1972:vi) postulated that the Windust Phase assemblages were evolutionarily descendent from the Lind Coulee assemblage, however this was from a time when the Lind Coulee site was thought to significantly predate the Windust era sites. Later dating firmly established a contemporaneous age for the Windust and Lind Coulee assemblages (WSU 2020). Recent findings at several early sites in the Pacific Northwest suggest that Haskett style points found at sites in Oregon (Paisley Cave, 35LK3400) and Idaho (Cooper's Ferry, 10IH73) likely represent the earliest diagnostic points of the WST (Brown et al 2019:487, Jenkins et al 2012). Haskett points found at Sentinel Gap (45KT1362), located some 200 miles downstream on the Columbia River from 45LI224, date to 11,900 cal BP (10,200 BP). However, it should also be noted that Lohse and Moser (2014:Figure 2) show Haskett style points as post-dating both Lind Coulee and Windust style points.

The majority of the projectile points and projectile point fragments observed at 45LI224 are examples of points typical of the Windust type series of the WST (Figure 1, Table 2). The complete point, 2015-006, is Windust A, the other complete point, 2015-001, is the only definitive Windust C point on the site. The nearly complete point fragment, 2017-116, is Windust B. The other Windust point base fragments are a mix of Windust A[3] (n=8) and Windust B[4] (n=6).

[3] Windust A base fragments: 2018-206, 2018-205, 2017-214, 2015-004, 2015-007, 2015-008, 2018-188, 2015-009.
[4] Windust B base fragments: 2018-059, 2017-009, 2015-010, 2017-119, 2018-089, 2018-121.

However, Windust type series projectile points are not the only points found at 45LI224, several (n=8) of the point base fragments appear to be more typical of Haskett, or even perhaps Lind Coulee style points[5], with steeply contracting stems and narrow convex bases, these are illustrated in the fourth row of Figure 1. These fragments all have contracting stems and narrow convex bases, elements that are present in both Haskett and Lind Coulee type points (Lohse and Moser 2014:Figure 2).

The dominance of basal, and basal and midsection, fragments in the collection indicate that the site occupants were rejuvenating darts for reuse and discarding and replacing points that were too damaged. The sheer number of discarded points at the site suggests that the site may have been used over an extended time frame, perhaps revisited seasonally over several years, or perhaps used once by a single large group.

Table 2. Diagnostic Projectile Points Collected From 45LI224, Sorted By Type

Catalog #	Artifact #	Description	Point Type
2015-001	N/A	Complete point, broken tip, no shoulder, contracting stem, concave base with slight "ears." CCS	Windust C
2015-006	N/A	Complete point, broken tip, distinct shoulders, contracting stem, slightly convex base. Basalt	Windust A
2015-004	N/A	Fragment, distinct shoulders, short contracting stem, straight base. CCS	Windust A
2015-007	N/A	Fragment, may have been reworked into a scraper. One shoulder, straight stem, straight to slightly convex base. CCS	Windust A
2015-008	N/A	Fragment, distinct shoulders, straight stem, straight to slightly convex base. CCS	Windust A
2015-009	N/A	Fragment, slightly contracting stem and straight base. CCS	Windust A
2017-214	214	Fragment, slightly contracting stem and convex base, similar to Lind Coulee point? CCS	Windust A/Lind Coulee?
2018-188	330	Fragment, midsection and stem, distinct shoulders, straight stem, straight base. CCS	Windust A
2018-205	347	Fragment, one distinct shoulder, slightly contracting stem, straight to slightly convex base. CCS	Windust A
2018-206	348	Fragment, one distinct shoulder, contracting stem, straight to slightly concave base. CCS	Windust A
2015-010	N/A	Fragment, straight stem with concave base. CCS	Windust B
2017-009	009	Fragment, contracting stem with concave base. CCS	Windust B
2017-119	119	Fragment, straight to slightly contracting stem with concave base. CCS	Windust B
2018-059	204	Fragment, midsection and base with distinct shoulders and short slightly contracting stem, concave base.	Windust B
2018-089	234	Fragment, straight to slightly expanding stem, slightly concave base. CCS	Windust B

[5] Haskett base fragments: 2015-002, 2017-031, 2017-025, 2017-060, 2017-085, 2018-057, 2017-175, 2018-197.

Catalog #	Artifact #	Description	Point Type
2018-121	266	Fragment, straight to slightly contracting stem, slightly concave base. CCS	Windust B
2015-002	N/A	Fragment, perhaps one shoulder, contracting stem with pointed convex base. CCS	Haskett/Lind Coulee
2017-025	25	Fragment, contracting stem with narrow convex base. CCS	Haskett/Lind Coulee
2017-031	31	Fragment, contracting stem with pointed convex base. Could be a point tip instead of a base. CCS	Haskett/Lind Coulee
2017-060	60	Fragment, contracting stem with narrow convex base. CCS	Haskett/Lind Coulee
2017-085	85	Fragment, contracting stem with narrow convex base. CCS	Haskett/Lind Coulee
2017-175	175	Fragment, contracting stem with narrow convex base. Could be a biface tip. CCS	Haskett/Lind Coulee
2018-057	202	Fragment, contracting stem with narrow convex base. CCS	Haskett/Lind Coulee
2018-197	339	Fragment, contracting stem with narrow convex base. CCS	Haskett/Lind Coulee

The wide variety of WST point styles represented in the 45LI224 assemblage is also of some interest. There are nearly equal numbers of Windust A, B, and Haskett style points, and a single Windust C, represented in the projectile points at 45LI224. There is no clear evidence in the projectile point assemblage of a post WST use of the site. Does this variety simply represent a diverse and varied set of tools simultaneously in use, or does it represent an evolutionary shift over time, suggesting a period of use at the site that spans the entirety of the WST? Chatters et al. (2012:43) describe that "…WST people were mobile foragers who created a diverse array of composite implements to use during a highly patterned but widely ranging subsistence round that emphasized big game," a notion, at least partially supported by the diverse projectile point assemblage at 45LI224.

The radiocarbon date of 8684 ± 93 cal BP (Beta Analytic Laboratory number 528368, Appendix B – Beta Analytic Radiocarbon dates and University of New Mexico Radiocarbon date) from sediment derived from the intact buried cultural horizon suggests a late WST use of the site. The only faunal fragment thus far recovered from the cultural horizon was subjected to minimally destructive radiocarbon dating. It has, so far, yielded two dates that are in conflict with both the sediment sample radiocarbon date and the typological time span of the projectile points from the site. The first bone date was 3830 ± 60 cal BP, the second date from the same bone was 4510 ± 60. Possible sources for error in these bone dates is discussed in the radiocarbon laboratory report, copied in Appendix B – Beta Analytic Radiocarbon dates and University of New Mexico Radiocarbon date.

The collection locations of the 2017 and 2018 stylistically diagnostic projectile points is depicted in Figure 4. As can be seen, all but one of these projectile points were observed on the gravel ridge area of the site in a displaced lagged out context. It is unknown if the observed location of these artifacts bears an association with their primary depositional location, and if so, to what extent; the distribution of projectile points by style seems to suggest it does not. All three identified point types are scattered and mixed across the gravel ridge area in a portion of the site where exposed, lagged gravel deposits are impacted by waves and reworked.

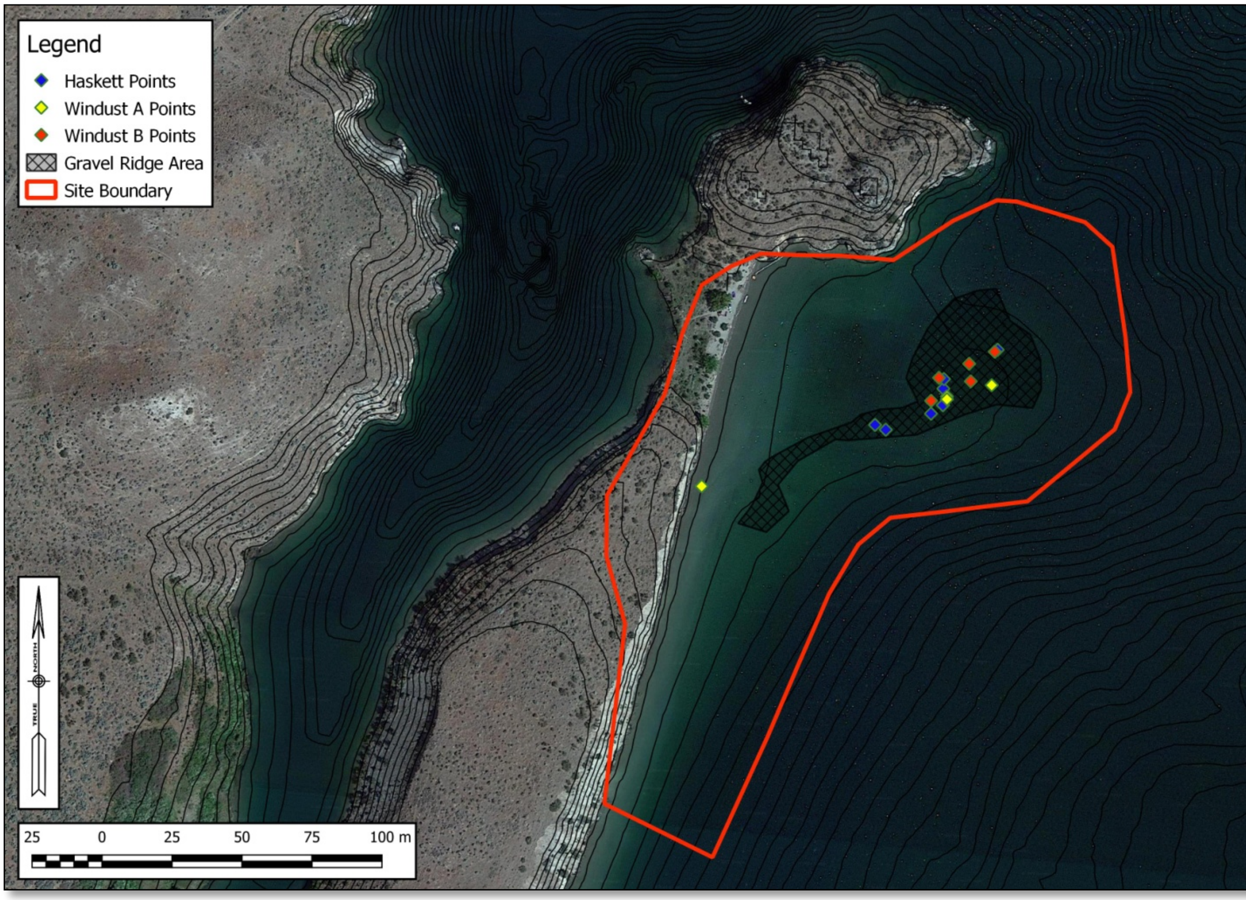

Figure 4. Distribution map of diagnostic projectile points collected from the 45LI224 site surface in 2017 and 2018 shown by projectile point type.

The projectile points thus far collected from 45LI224 indicate the site is one of a handful of rare, early sites in Washington, and the only confirmed/accepted WST site along this section of the Columbia River. The projectile point assemblage at the site contains all of the points that are identified as forming the WST assemblage in the Columbia Plateau. Intact cultural deposits remaining at the site add context to artifacts displaced by reservoir induced erosion, and have provided an initial radiocarbon date to confirm the antiquity of the site. As stated earlier, this is just a preliminary examination of the projectile point assemblage collected from 45LI224, a thorough and comprehensive analysis of the entire lithic assemblage of the site should be conducted.

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Appendix A – Artifact photographs

Figure 5. 45LI224, Windust style projectile point (CAT# 45LI224 2015-001), collected from the site in April 2015 (adapted from Cheung 2015:13 Figure 6).

Figure 6. Stemmed argillite lanceolate projectile point (CAT# 45LI224 2015-006) observed in the drawdown zone at 45LI224 in May 2015. 2015 Site Condition Monitoring photo #235.

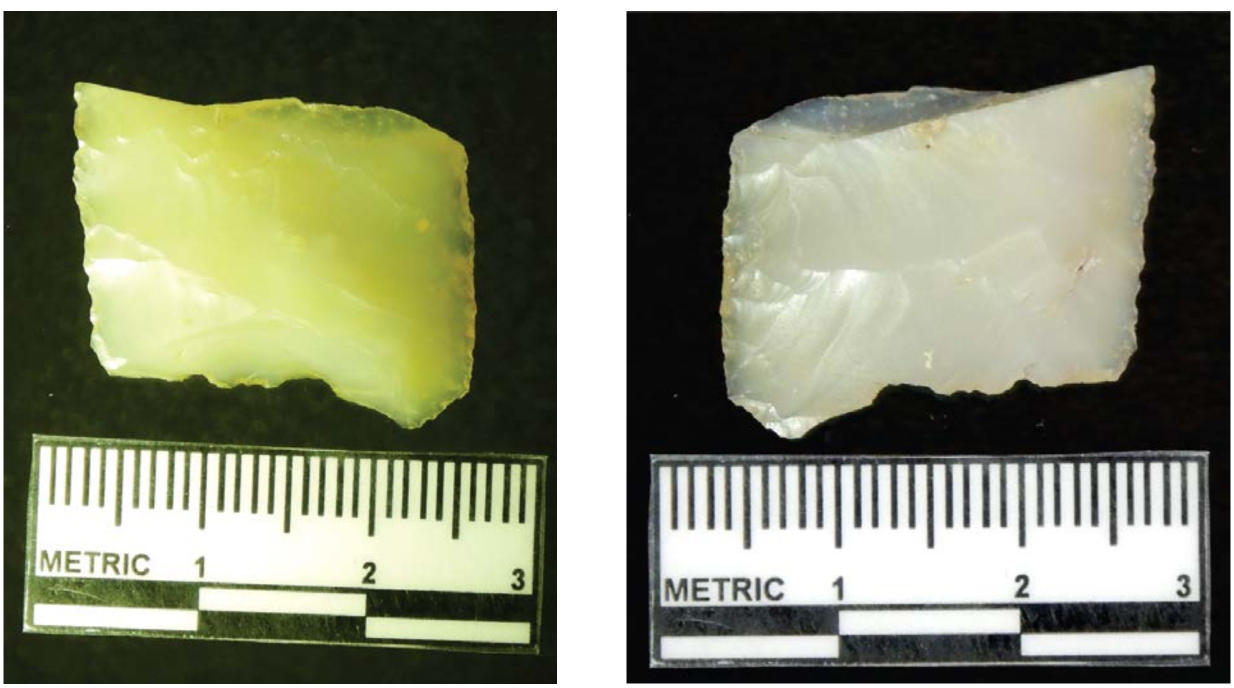

Figure 7. Projectile point midsection (CAT# 45LI224 2018-215) observed in the drawdown zone at 45LI224 in May 2018. Translucent, white, cryptocrystalline silicate projectile point midsection measuring 22 mm tall by 23 mm wide.

Appendix B – Beta Analytic Radiocarbon dates and University of New Mexico Radiocarbon date

BETA Beta Analytic TESTING LABORATORY

Beta Analytic Inc
4985 SW 74 Court
Miami, Florida 33155
Tel: 305-667-5167
Fax: 305-663-0964
info@betalabservices.com

ISO/IEC 17025:2005-Accredited Testing Laboratory

June 26, 2019

Mr. Guy Moura
Confederated Tribes of the Colville Reservation
P.O. Box 150
Nespelem, WA 99155
United States

RE: Radiocarbon Dating Results

Dear Mr. Moura,

Enclosed is the radiocarbon dating result for one sample recently sent to us. As usual, specifics of the analysis are listed on the report with the result and calibration data is provided where applicable. The Conventional Radiocarbon Age has been corrected for total fractionation effects and where applicable, calibration was performed using 2013 calibration databases (cited on the graph pages).

The web directory containing the table of results and PDF download also contains pictures, a cvs spreadsheet download option and a quality assurance report containing expected vs. measured values for 3-5 working standards analyzed simultaneously with your samples.

The reported result is accredited to ISO/IEC 17025:2005 Testing Accreditation PJLA #59423 standards and all pretreatments and chemistry were performed here in our laboratories and counted in our own accelerators here in Miami. Since Beta is not a teaching laboratory, only graduates trained to strict protocols of the ISO/IEC 17025:2005 Testing Accreditation PJLA #59423 program participated in the analysis.

As always Conventional Radiocarbon Ages and sigmas are rounded to the nearest 10 years per the conventions of the 1977 International Radiocarbon Conference. When counting statistics produce sigmas lower than +/- 30 years, a conservative +/- 30 BP is cited for the result. The reported d13C was measured separately in an IRMS (isotope ratio mass spectrometer). It is NOT the AMS d13C which would include fractionation effects from natural, chemistry and AMS induced sources.

When interpreting the result, please consider any communications you may have had with us regarding the sample. As always, your inquiries are most welcome. If you have any questions or would like further details of the analysis, please do not hesitate to contact us.

Our invoice will be emailed separately. Please forward it to the appropriate officer or send a credit card authorization. Thank you. As always, if you have any questions or would like to discuss the results, don't hesitate to contact us.

Sincerely,

Chris Patrick
Digital signature on file

Chris Patrick Director

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Beta Analytic
TESTING LABORATORY

Beta Analytic Inc
4985 SW 74 Court
Miami, Florida 33155
Tel: 305-667-5167
Fax: 305-663-0964
info@betalabservices.com

ISO/IEC 17025:2005-Accredited Testing Laboratory

REPORT OF RADIOCARBON DATING ANALYSES

Guy Moura		Report Date:	June 26, 2019
Confederated Tribes of the Colville Reservation		Material Received:	June 17, 2019

Laboratory Number	Sample Code Number	Conventional Radiocarbon Age (BP) or Percent Modern Carbon (pMC) & Stable Isotopes Calendar Calibrated Results: 95.4 % Probability High Probability Density Range Method (HPD)	
Beta - 528368	**45LI224 2017 TU2 Cat 211**	**7880 +/- 30 BP**	IRMS δ13C: -23.9 o/oo

(94.0%)	**6828 - 6642 cal BC**	**(8777 - 8591 cal BP)**
(1.4%)	**6907 - 6888 cal BC**	**(8856 - 8837 cal BP)**

Submitter Material:	Organic Sediment/Gyttja
Pretreatment:	(organic sediment) acid washes
Analyzed Material:	Organic sediment
Analysis Service:	AMS-Standard delivery
Percent Modern Carbon:	37.49 +/- 0.14 pMC
Fraction Modern Carbon:	0.3749 +/- 0.0014
D14C:	-625.05 +/- 1.40 o/oo
Δ14C:	-628.17 +/- 1.40 o/oo(1950:2,019.00)
Measured Radiocarbon Age:	(without d13C correction): 7860 +/- 30 BP
Calibration:	BetaCal3.21: HPD method: INTCAL13

Results are ISO/IEC-17025:2005 accredited. No sub-contracting or student labor was used in the analyses. All work was done at Beta in 4 in-house NEC accelerator mass spectrometers and 4 Thermo IRMSs. The "Conventional Radiocarbon Age" was calculated using the Libby half-life (5568 years), is corrected for total isotopic fraction and was used for calendar calibration where applicable. The Age is rounded to the nearest 10 years and is reported as radiocarbon years before present (BP), "present" = AD 1950. Results greater than the modern reference are reported as percent modern carbon (pMC). The modern reference standard was 95% the 14C signature of NIST SRM-4990C (oxalic acid). Quoted errors are 1 sigma counting statistics. Calculated sigmas less than 30 BP on the Conventional Radiocarbon Age are conservatively rounded up to 30. d13C values are on the material itself (not the AMS d13C). d13C and d15N values are relative to VPDB-1. References for calendar calibrations are cited at the bottom of calibration graph pages.

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BetaCal 3.21

Calibration of Radiocarbon Age to Calendar Years

(High Probability Density Range Method (HPD): INTCAL13)

(Variables: d13C = -23.9 o/oo)

Laboratory number **Beta-528368**

Conventional radiocarbon age **7880 ± 30 BP**

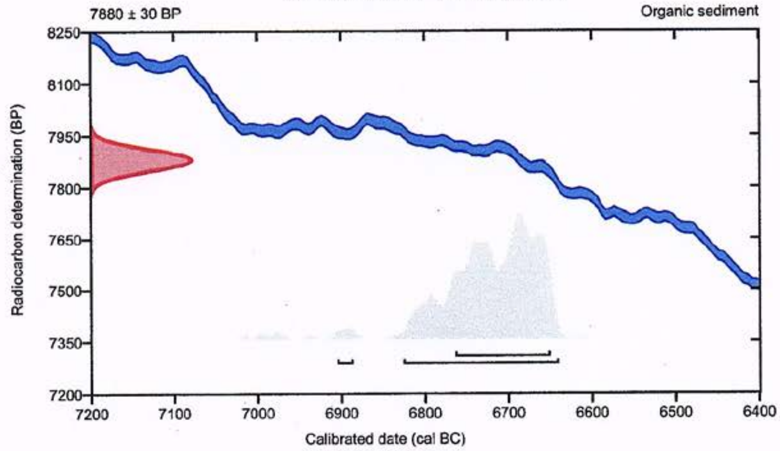
95.4% probability

(94%)	6828 - 6642 cal BC	(8777 - 8591 cal BP)
(1.4%)	6907 - 6888 cal BC	(8856 - 8837 cal BP)

68.2% probability

(68.2%)	6766 - 6652 cal BC	(8715 - 8601 cal BP)

45LI224 2017 TU2 Cat 211



Database used
INTCAL13

References

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References to Database INTCAL13
Reimer, et.al., 2013, Radiocarbon55(4).

Beta Analytic Radiocarbon Dating Laboratory
4985 S.W. 74th Court, Miami, Florida 33155 • Tel: (305)667-5167 • Fax: (305)663-0964 • Email: beta@radiocarbon.com
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Beta Analytic Inc
4985 SW 74 Court
Miami, Florida 33155
Tel: 305-667-5167
Fax: 305-663-0964
info@betalabservices.com

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Quality Assurance Report

This report provides the results of reference materials used to validate radiocarbon analyses prior to reporting. Known-value reference materials were analyzed quasi-simultaneously with the unknowns. Results are reported as expected values vs measured values. Reported values are calculated relative to NIST SRM-4990B and corrected for isotopic fractionation. Results are reported using the direct analytical measure percent modern carbon (pMC) with one relative standard deviation. Agreement between expected and measured values is taken as being within 2 sigma agreement (error x 2) to account for total laboratory error.

Report Date: June 26, 2019
Submitter: Mr. Guy Moura

QA MEASUREMENTS

Reference 1

Expected Value: 0.42 +/- 0.04
Measured Value: 0.43 +/- 0.02 pMC
Agreement: Accepted

Reference 2

Expected Value: 96.69 +/- 0.50 pMC
Measured Value: 96.77 +/- 0.29 pMC
Agreement: Accepted

Reference 3

Expected Value: 129.41 +/- 0.06 pMC
Measured Value: 129.44 +/- 0.37 pMC
Agreement: Accepted

COMMENT: All measurements passed acceptance tests.

Validation: Chris Patrick
Digital signature on file

Date: June 26, 2019

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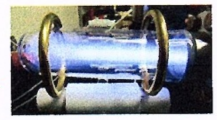

Radiocarbon Sampling and Dating Report – August 12, 2020

Brenda Covington | Archaeologist
History/Archaeology Program
Confederated Tribes of the Colville Reservation
P.O. Box 150 | Nespelem, WA 99155

Client Sample Designation: 45LI224 2017.204

OAS Sample Label (used for internal records): Bone 204

OAS CO_2 Sample Number(s): 190805c-1; 190806c-1

ETH AMS Sample Number(s): 103876.1; 104589.1

Dating Results:

Sample number			Radiocarbon age		$\delta^{13}C$
ETH	OAS Label	OAS	Years BP	σ (± years)	‰
103876.1	Bone 204	190805c-1	3,830	60	-18.6
104589.1	Bone 204	190806c-1	4,510	60	-19.7

Discussion:

The specimen was photographed as received (Figure 1) and was cleaned with six distilled water rinses with ultrasonication, loosening and removing adhering sediment. There was no cloudiness after the sixth cleaning, and the bone was dried in a 40°C oven and re-photographed (Figure 2). The specimen was then rinsed and ultrasonicated twice to saturate the bone with distilled water, and it was placed in 1 ml of phosphate buffer solution (pH 8) and ultrasonicated. If humic acid contamination is present in a specimen, the clear solution turns brown to pale yellow, depending on the amount of contamination. In this case, the solution remained clear, indicating no apparent contamination. The specimen was rinsed twice in distilled water then ultrasonicated twice in distilled water to remove absorbed phosphate buffer solution. The specimen was placed on a porcelain support and dried in preparation for plasma oxidation (Figure 3).

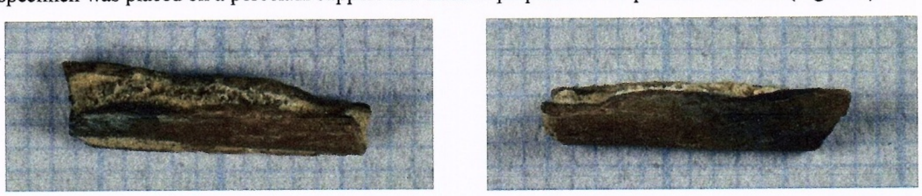

Figure 1. Bone 204 as received at OAS. Image background is millimeter graph paper.

Plasma oxidation at the OAS laboratory is described in detail in Rowe et al. 2017. A significant advantage of this approach is that plasma oxidation occurs at energy levels below the dissociation threshold of carbonates. The carbon composition of carbonates therefore does not contribute to the radiocarbon sample, and no pretreatment is required for carbonate contaminant removal.

Center for New Mexico Archaeology
7 Old Cochiti Road, Santa Fe, New Mexico 87507-1004
Phone: (505) 476-4401 Fax: (505) 476-4448

Office of Archaeological Studies
www.nmarchaeology.org

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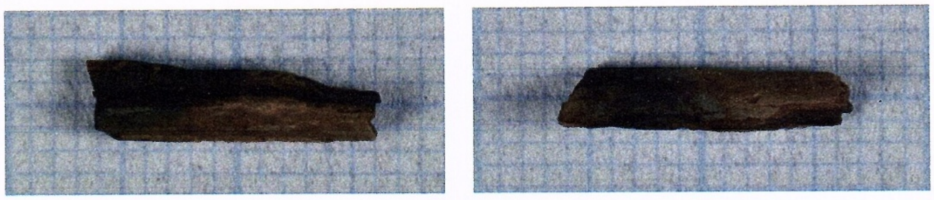

Figure 2. Bone 204 after cleaning with distilled water.

Figure 3. Bone 204 in a porcelain support prior to plasma oxidation sampling.

The specimen was placed in a vacuum chamber which was then evacuated to a pressure of circa 10^{-6} Torr (high vacuum). Low pressure argon was introduced, and the specimen was bathed in a low-energy argon plasma (100 watts of radio frequency [RF] energy, resulting in a within-chamber temperature of circa 108° C). The argon plasma scours the chamber and specimen surfaces, knocking free any "sticky" atmospheric CO_2 molecules that would be modern contamination during radiocarbon dating. By itself, an argon plasma will not initiate chemical oxidation. However, the RF energy and the warmth of the argon plasma can cause the release of bound water in specimens, and the combined argon-water plasma can initiate some oxidation. In the case of Bone 204, system pressure changes suggested that it is likely that water release and argon-water oxidation did occur during the first argon plasma, evolving as much as 100 µg of carbon in the form of CO_2 from both sticky and oxidized sources.

Negligible CO_2 (<0.1 µg of carbon) was detected after the third argon plasma cleaning, and after evacuation to 10^{-6} Torr, low pressure oxygen was introduced. Plasma energy was held at 10 watts for 50 minutes, and approximately 100 µg of carbon were collected for dating (190805c-1). After collection and evacuation, a second charge of low pressure oxygen was introduced to the chamber. A15 watt plasma was maintained for 45 minutes, and approximately 98 µg of carbon were collected as a second dating sample (190806c-1).

The specimen showed slight bleaching when it was removed from the sampling chamber after the two plasma oxidations (Figure 4).

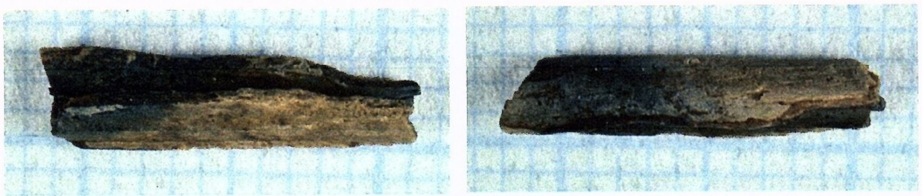

Figure 4. Bone 204 after plasma exposure.

The first sample collected was submitted to ETH, Zurich, for direct AMS dating of the carbon dioxide. The result (3,830±60) was younger than the anticipated age of early Archaic or older. The calibrated result is presented in Figure 5.

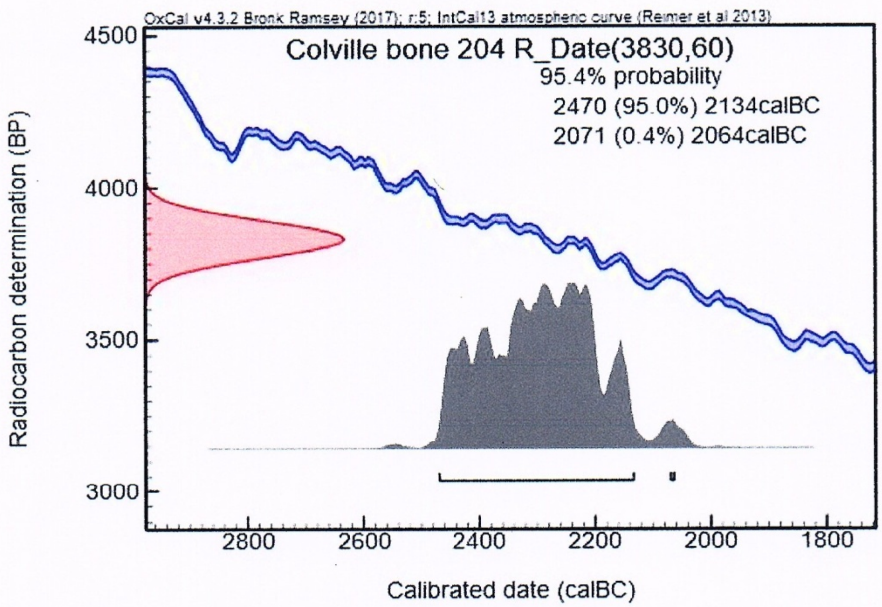

Figure 5. Tree-ring calibration of the 3,830±60 BP radiocarbon age for the first sample of Bone 204 (190805c-1, ETH 103876.1).

Due to the disparity between expected and actual dating results, the second CO_2 sample was then submitted to ETH for AMS isotope measurement and dating. Its age is older (Figure 6) and is sufficiently older (4,510±60 BP) that the difference between the two dates is beyond what could be expected from measurement error if two separate samples had been collected from specimen carbon of a single age.

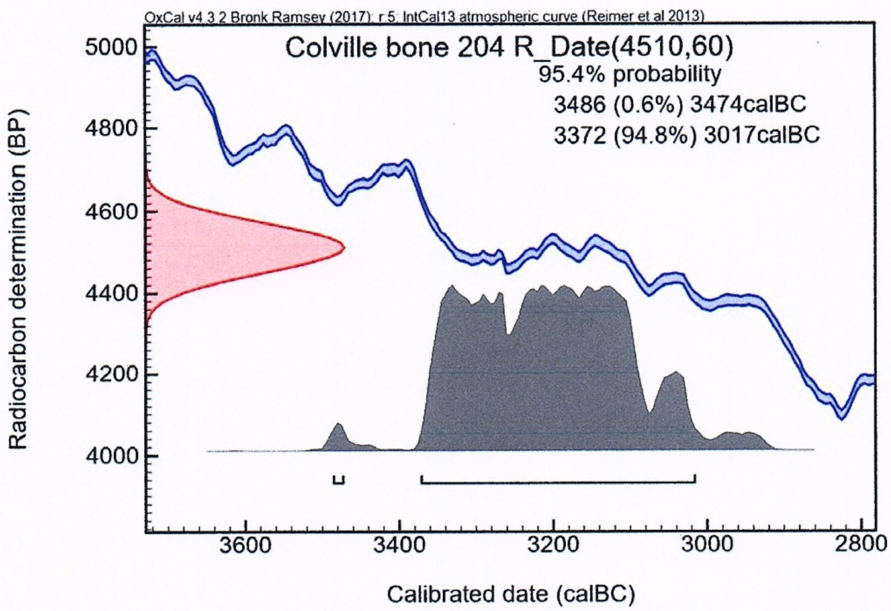

Figure 6. Tree-ring calibration of the 4,510±60 BP radiocarbon age for the second sample of Bone 204 (190805c-1, ETH 104589.1).

The younger age from the initial CO_2 extraction (sample 190805c-1) could result from modern carbon contamination of the bone surface compared to the second CO_2 extraction (sample 190806c-1). To estimate the magnitude of the possible "modern 1950 contamination" of carbon of the specimen, we use the modified approach of Mook and Waterbolk (1985:27-28). This approach calculates contamination in terms of fractional ^{14}C activity.

$$X = (F^{14}C_{\text{"contaminated sample"}} - F^{14}C_{\text{" no contamination"}}) / (F^{14}C_{\text{"contamination source"}} - F^{14}C_{\text{" no contamination"}}) \quad (1)$$

where $F^{14}C_{\text{"contamination source"}} = F^{14}C_{\text{"modern"}} = 1.0$

or where $F^{14}C_{\text{"contamination source"}} = F^{14}C_{\text{"dead"}} = 0$

To calculate the fractional ^{14}C activity use the fundamental radiocarbon decay equation

$$F^{14}C = e^{-(T/8033)} \quad (2)$$

where $F^{14}C$ is the fractional ^{14}C activity of a given date in BP and T is a given date in years BP

Using this approach, we calculated the theoretical effect of modern carbon contamination on Bone 204, by assuming the date on sample 190805c-1 resulted from modern CO_2 contamination of the date on CO_2 sample 190806c-1.

Assumptions for calculation:

190805c-1 is the CO_2 sample *assumed* to be contaminated with modern carbon because it is both the first extracted and the younger of the two. Then $T_{\text{"contaminated sample"}}$ = 3830 ^{14}C years BP (age measured at the ETH-AMS laboratory from CO_2 sample extracted at OAS), $F^{14}C_{\text{"contaminated sample"}}$ is calculated according to equation (2) = 0.6208 which is the value given by AMS lab as $F^{14}C_{\text{"190805c-1"}}$.

190806c-1 is the CO_2 sample *assumed* to be uncontaminated by modern carbon because it is older of the two. Then $T_{\text{" no contamination"}}$ = 4510 ^{14}C years BP (age given by AMS AMS laboratory from CO_2 sample extracted at OAS), $F^{14}C_{\text{" no contamination "}}$ is calculated according to equation (2) to be 0.5704 which is the value given by AMS lab as $F^{14}C_{\text{"190806c-1"}}$

With these two assumptions,

$X = (F^{14}C_{\text{"contaminated sample"}} - F^{14}C_{\text{" no contamination"}}) / (F^{14}C_{\text{"contamination source"}} - F^{14}C_{\text{" no contamination"}})$

$X = (F^{14}C_{\text{"190805c-1"}} - F^{14}C_{\text{"190806c-1"}}) / (F^{14}C_{\text{"modern contamination"}} - F^{14}C_{\text{"190806c-1"}})$

$X = (0.6208 - 0.5704) / (1.0 - 0.5704)$

$X = 0.1173$

$\mathbf{X_{\text{as a percent}}}$ **= 11.7%**, i.e., CO_2 sample 1908905c-1 would be composed of 88.3% CO_2 with the age 4510 BP and 11.7% modern contamination. So to get the younger age (assuming the older age were the true age) there would have to be about 12 percent modern carbon contamination.

If the specimen were contaminated only since excavation, then the calculation assuming $F^{14}C_{\text{"contamination modern"}}$ = 1.0 would hold true. Contamination could be in the form of either absorbed modern carbon dioxide within the bone's pore space or modern exchanged carbon within the organic molecular structure of the specimen. Our laboratory protocols minimize risk of any pore space contamination by running argon plasmas and letting the bone sit under high vacuum conditions for hours to days. This has been shown to be effective in other test cases. Potential contamination by molecular exchange is unlikely due to the careful handling and short time since excavation.

A more likely mechanism of contamination is that the molecular structure of the bone has exchanged some its carbon with ambient environmental carbon over the entire period from post-death to present. In this case, the assumption $F^{14}C_{\text{"contamination modern"}}$ = 1.0 does NOT hold true and instead the value $F^{14}C_{\text{"contamination"}}$ would be less than the 1.0 used to calculate X. As a result, the fraction of contamination with ambient carbon (X) is likely greater than the reported 12 percent.

When applied to intact specimens, plasma oxidation is a surface biased radiocarbon sampling technique. Plasma oxidation sampling has documented the changing isotopic composition progressively within the mass of Bone 204. Since the first sample is younger than the second, surface diffusion of the contaminating carbon is implied by these dating results. Another possible interpretation of how plasma oxidation works is that it is preferentially sampling carbon from easily broken organic molecular bonds. If substitution happens at weak bond positions, the contaminating carbon may be oxidized preferentially, increasing the proportion of contaminating carbon in the first plasma oxidation. Progressive sampling would exhaust the weakly bonded contaminating carbon, and later plasma samples would be dominated by the carbon incorporated into stronger molecular structures. Assuming that the contamination source is also surface-biased and/or also is reflected in weak molecular structures, progressive sampling and dating should reflect a continuum of more-to-less contaminated carbon content.

Of the two dates reported here for the specimen, we believe that the older date is a better approximation of the age of Bone 204. However, based on either the surface or weak bond model of contamination, the true age of the animal at time of death is probably even older.

Eric Blinman, Marvin Rowe, and Shelby A. Jones

References

Mook, W.G. and H.T. Waterbolk (1985) *Handbooks for Archaeologists: No. 3, Radiocarbon Dating*, European Science Foundation, Strasbourg, pp. 27-28.

Rowe M.W., E. Blinman, J,C, Martin, J.R. Cox, M. MacKenzie and L. Wacker (2017) Cold plasma oxidation and "nondestructive" radiocarbon sampling. In T. Britt, ed., *Proceedings of the Twenty-year retrospective of National Center for Preservation Technology and Training*. Natchitoches, LA: National Center for Preservation Technology and Training, ISBN:electronic 978-9970440-3-4, pp. 64-77.