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Author(s) / Editor(s): Peter Kovacik, Linda Scott Cummings
Published: PRI Technical Report ,2014-006. 2014
Document Type: book
Stable URL: https://core.tdar.org/document/380486/phytolith-and-macrofloral-analysis-and-ams-radiocarbon-dating-from-trinity-oracel-pipeline-1720-la121520-eddy-county-new-mexico
DOI: doi:10.6067/XCV8S46RJT

Note: 2 year embargo

PHYTOLITH AND MACROFLORAL ANALYSIS AND AMS RADIOCARBON DATING FROM TRINITY-ORACEL PIPELINE #1720, LA121520, EDDY COUNTY, NEW MEXICO

By

Peter Kováčik
and
Linda Scott Cummings

With assistance from
R. A. Varney

PaleoResearch Institute
Golden, Colorado

PaleoResearch Institute Technical Report 14-006

Prepared for

Lone Mountain Archaeological Services, Inc.
Albuquerque, New Mexico

April 2014

INTRODUCTION

Fill that included carbon-stained sediments from a fire-cracked rock concentration at site LA 121520 in Eddy County, southern New Mexico, was submitted for phytolith and macrofloral analysis. Charcoal recovered from the macrofloral portion of the sample was submitted for AMS radiocarbon age determination. The sample is expected to date to the historic period.

METHODS

Phytoliths

Extraction of phytoliths from this sample was based on heavy liquid floatation. Sodium hypochlorite (bleach) was first used to destroy the organic fraction from 15 ml of sediment. Once this reaction was complete, the sample was rinsed to remove the bleach. Because the sample was expected to contain calcium carbonates, it was reacted with 30% hydrochloric acid, then rinsed until neutral. A small quantity of sodium hexametaphosphate was added once the sample reached neutrality, then it was allowed to settle according to Stoke's Law in a 600 ml beaker. This process was repeated with EDTA. These steps remove clay particles prior to heavy liquid separation. Next, the sample was freeze dried. The dried silts and sands were then mixed with sodium polytungstate (density 2.3) and centrifuged to separate the phytoliths, which will float, from the other silica, which will not. Phytoliths, in the broader sense, include opal silica phytoliths and calcium oxalate crystals. Calcium oxalate crystals are formed by *Opuntia* (prickly pear cactus),*Yucca* (yucca), and other plants. Unfortunately, these forms rarely survive in sediments. Any remaining clay was floated with the phytoliths and was further removed by centrifugation. The sample was then rinsed with distilled water, then alcohols to remove the water. After several alcohol rinses, the sample was mounted in immersion oil for counting with a light microscope at a magnification of 500x, then scanned using cross-polar illumination. The phytolith diagram was produced using Tilia, a computer program developed by Dr. Eric Grimm of the Illinois State Museum for diagraming pollen.

Macrofloral

The macrofloral sample was floated using a modification of the procedures outlined by Matthews (1979). The sample was added to approximately three gallons of water, then stirred until a strong vortex formed. The floating material (light fraction) was poured through a 150-micron-mesh sieve. Additional water was added and the process repeated until all floating material was removed from the sample (a minimum of five times). The material that remained in the bottom (heavy fraction) was poured through a 0.5-mm-mesh screen. The floated portions were allowed to dry.

The light fraction was weighed, then passed through a series of graduated screens (US Standard Sieves with 4-mm, 2-mm, 1-mm, 0.5-mm, and 0.25-mm openings) to separate charcoal debris and to initially sort the remains. The contents of each screen then were examined. Charcoal pieces larger than 0.5 mm in diameter were separated from the rest of the light fraction, and the total charcoal was weighed. Charcoal pieces in a representative sample were broken to expose fresh cross, radial, and tangential sections, then examined under a

binocular microscope at a magnification of 70x and under a Nikon Optiphot 66 microscope at magnifications of 320–800x. The weights of each charcoal type within the representative sample were recorded. The material that remained in the 4-mm, 2-mm, 1-mm, 0.5-mm, and 0.25-mm sieves was scanned under a binocular stereo microscope at a magnification of 10x, with some identifications requiring magnifications of up to 70x. The material that passed through the 0.25-mm screen was not examined. The heavy fraction was scanned at a magnification of 2x for the presence of botanic remains. Estimates of frequencies were calculated from a portion of the total volume floated and are noted in the macrofloral table with an asterisk (*). The term "seed" is used to represent seeds, achenes, caryopses, and other disseminules. Remains from the light and heavy fractions were recorded as charred and/or uncharred, whole and/or fragments. Macrofloral remains, including charcoal, were identified using manuals (Carlquist 2001; Hoadley 1990; Martin and Barkley 1961; Musil 1963; Schopmeyer 1974) and by comparison with modern and archaeological references.

Samples from archaeological sites commonly contain both charred and uncharred remains. Many ethnobotanists use the basic rule that unless there is a specific reason to believe otherwise, only charred remains will be considered prehistoric (Minnis 1981:147). Minnis (1981:147) states that it is "improbable that many prehistoric seeds survive uncharred through common archaeological time spans." Few seeds survive longer than a century, and most survive for a much shorter period of time (Harrington 1972; Justice and Bass 1978; Quick 1961). It is presumed that once seeds have died, decomposing organisms work to decay the seeds. Sites with unusual levels of preservation, such as caves, water-logged areas, and very arid areas, however, can contain uncharred prehistoric remains. The question of whether an uncharred seed represents presence in the prehistoric record is considered on a sample-by-sample basis. Extraordinary conditions for preservation are required.

AMS Radiocarbon Dating - Charcoal

The charcoal sample submitted for radiocarbon dating was identified and weighed prior to selecting a subsample for pre-treatment. The remainder of the subsample that proceeds to pre-treatment, if any, is permanently curated at PaleoResearch. The subsample selected for pre-treatment first was freeze-dried using a vacuum system, which freezes out all moisture at -107 °C and < 10 millitorr. The sample then was subjected to hot (at least 110 °C), 6N hydrochloric acid (HCl), with rinses to neutral between each HCl treatment, until the supernatant was clear. This step removes iron compounds and calcium carbonates that hamper removal of humate compounds. Next, the sample was subjected to 5% potassium hydroxide (KOH) to remove humates. Once again, the sample was rinsed to neutral and re-acidified with pH 2 HCl between each KOH step. This step was repeated until the supernatant was clear, signaling removal of all humates. After humate removal, the sample was freeze-dried, then combined with cupric oxide (CuO) and elemental silver (Ag) in a quartz tube in a ratio based on the mass of carbon in the sample. The tube was hydrogen flame-sealed under vacuum.

Standards and laboratory background wood samples were simultaneously treated to the same acid and base processing as the charcoal sample of unknown age. A radiocarbon “dead” wood blank from the Grey Fossil site in Washington County, Tennessee, that is believed to date to the Hemphillian stage of the late Miocene, 4.5–7 MYA (currently beyond the detection capabilities of AMS) was used to calibrate the laboratory correction factor. Standards of known age, such as TIRI Sample “B” (Belfast Pine) with a consensus age of 4503 ± 6, and TIRI

Sample “J” (Bulston Crannog wood) with a consensus age of 1605 ± 8 (Gulliksen and Scott 1995), also were used to establish the laboratory correction factor. Each wood standard was run in a quantity similar to the submitted samples of unknown age and sealed in a quartz tube after the requisite pre-treatment. Once all the wood standards, blanks, and submitted samples of unknown age were prepared and sealed in their individual quartz tubes, they were combusted at 820 °C, soaked for an extended period of time at that temperature, and then slowly allowed to cool to enable the chemical reaction that extracts carbon dioxide (CO_2) gas.

Following this last step, the charcoal sample of unknown age, the wood standards, and the laboratory backgrounds were sent to the Center for Applied Isotope Studies in Athens, Georgia, where the CO_2 gas was processed into graphite. The graphite in these samples then was placed in the target and run through the accelerator, which produces the numbers that are converted into the radiocarbon date presented in the data section. Dates are presented as conventional radiocarbon ages, as well as calibrated ages using Intcalc04 curves on Oxcal version 3.10 (Bronk Ramsey 2005; Reimer et al. 2009). This is a probability-based method for determining conventional ages and is preferred over the intercept-based alternative because it provides a calibrated date that reflects the probability of its occurrence within a given distribution (reflected by the amplitude [height] of the curve), as opposed to individual point estimates. As a result, the probability-based method offers more stability to the calibrated values than those derived from intercept-based methods that are subject to adjustments in the calibration curve (Telford et al. 2004).

RADIOCARBON REVIEW

When interpreting radiocarbon dates from non-annuals such as trees and shrubs, it is important to understand that a radiocarbon date reflects the age of that portion of the tree/shrub when it stopped exchanging carbon with the atmosphere, not necessarily the date that the tree/shrub died or was burned. Trees and shrubs grow bigger each year from the cambium, where a new layer or ring of cells is added each year. During photosynthesis, new cells take in atmospheric carbon dioxide, which includes radiocarbon. The radiocarbon taken in will reflect the radiocarbon present in the atmosphere during that season of growth. Once the sapwood in a tree has been converted into heartwood, the metabolic process stops for that inner wood. Once this happens, no new carbon atoms are acquired, and the radiocarbon that is present starts to decay. Studies have shown that there is little to no movement of carbon-bearing material from one ring to another. As a result, wood from different parts of the tree will yield different radiocarbon dates. The outer rings exhibit an age close to the cutting or death date of the tree, while the inner rings will reflect the age of the tree. Because the younger, outer rings burn off first when a log or branch is burned, it is the older, inner rings that typically are what is left remaining in a charcoal assemblage (Puseman 2009; Taylor 1987).

PHYTOLITH REVIEW

Phytoliths are silica bodies produced by plants when soluble silica in the ground water is absorbed by the roots and carried up to the plant via the vascular system. Evaporation and metabolism of this water result in precipitation of the silica in and around the cellular walls. Opal

phytoliths, which are distinct and decay-resistant plant remains, are deposited in the soil as the plant or plant parts die and break down. They are, however, subject to mechanical breakage and erosion and deterioration in high pH soils. Phytoliths are usually introduced directly into the soils in which the plants decay. Transportation of phytoliths occurs primarily by animal consumption, gathering of plants by humans, or by erosion or transportation of the soil by wind, water, or ice. Phytoliths produced in roots/tubers will deteriorate at the level of those roots/tubers and will not be represented on the growing surface. Therefore, recovery of phytoliths representing roots/tubers from stratigraphic sediments do not necessarily represent vegetation coeval with that represented by phytoliths produced in leaves or other above-ground vegetative parts.

The three major types of grass short-cell phytoliths include festucoid, chloridoid, and panicoid. Smooth elongate phytoliths are of no aid in interpreting either paleoenvironmental conditions or the subsistence record, because they are produced by all grasses. Phytoliths tabulated to represent "total phytoliths" include the grass short-cells, buliform, trichome, elongate, and dicot forms. Frequencies for all other bodies recovered are calculated by dividing the number of each type recovered by the "total phytoliths".

The festucoid class of phytoliths is ascribed primarily to the subfamily Pooideae and occur most abundantly in cool, moist climates. However, Brown (1984) notes that festucoid phytoliths are produced in small quantity by nearly all grasses (mostly rondel-type phytoliths). Therefore, while they are typical phytoliths produced by the subfamily Pooideae, they are not exclusive to this subfamily. Chloridoid phytoliths (short saddles) are found primarily in the subfamily Chloridoideae, a warm-season grass that grows in arid to semi-arid areas and require less available soil moisture. Chloridoid grasses are the most abundant in the American Southwest (Gould and Shaw 1983:120). Bilobates and polylobates (lobates) are produced mainly by panicoid grasses, although a few of the festucoid grasses also produce these forms. Panicoid phytoliths occur in warm-season or tall grasses that frequently thrive in humid conditions. Twiss (1987:181) also notes that some members of the subfamily Chloridoideae produce both bilobate (panicoid) and festucoid phytoliths. “According to Gould (1983:110) more than 97% of the native US grass species (1,026 or 1,053) are divided equally among three subfamilies Pooideae, Chloridoideae, and Panicoideae" (Twiss 1987:181).

Buliform phytoliths are produced by grasses in response to wet conditions and are to be expected in wet habitats of floodplains and other places. Trichomes represent silicified hairs, which may occur on the stems, leaves, and the glumes or bran surrounding grass seeds.

Conifers produce opal silica phytoliths in their inner bark and needles. Polyhedral phytoliths are reported to be observed in leaves (Bozarth 1993), and at PaleoResearch Institute we have observed the blocky forms in bark reference samples.

DISCUSSION AND CONCLUSIONS

Site LA 121520, located in Eddy County, New Mexico, consists of four features that contained various historic artifacts. Local vegetation in the region of the site includes creosote bush (*Larrea tridentata*), mesquite (*Prosopis*), yucca (*Yucca*), and different grasses (Poaceae). Soil sample 1, collected at a depth of 0-10 cmbs from Feature 4, was examined for phytolith and

macrofloral remains (Table 1). In addition, suitable charred material recovered from this sample was submitted for AMS radiocarbon dating.

The macrofloral portion of sample 1 yielded a variety of uncharred floral remains (fruits, florets, seeds, and leaves) reflecting modern plants (Tables 2 and 3) including *Celtis* (hackberry), *Chenopodium* (goosefoot), Euphorbiaceae (spurge family), *Chamaesyce* (sandmat), *Euphorbia* (spurge), Fabaceae (bean family), *Prosopis* (mesquite), *Larrea tridentata* (creosote bush), Paniceae (panicgrass/bristlegrass), Solanaceae (nightshade family), *Trianthema portulacastrum* (desert horse purslane). Charred remains for this sample are represented only by very small charcoal fragments, including vitrified conifer, *Prosopis*, and vitrified unidentified hardwood fragments, reflecting a conifer wood, mesquite, and other hardwood wood that was too vitrified for further identification. Vitrified charcoal has a shiny, glassy appearance that can range from still recognizable in structure "to a dense mass, completely 'molten' and non-determinable" (Marguerie and Hunot 2007 in McParland, 2010 #6975). Although vitrification of charcoal has been attributed to burning at high temperature and/or burning green wood, the process of vitrification is not completely understood. Experimental studies and reflectance measurements on archaeological charcoal suggest that vitrification can occur at low temperatures. McParland *et al*. (2010) were unable to produce vitrification during experimental high temperature burning or by burning green wood. They suggest it is associated with post-depositional factors. If any consensus exists concerning vitrification, it is that more studies need to be conducted.

Fragments of mesquite charcoal were submitted for AMS radiocarbon age determination, returning a date of 123 ± 21 RCYBP (PRI-14-006-1). This date hits a relatively flat part of the calibration curve that exhibits minimal oscillation, producing a broad calibrated age range of 270–180 and 150–10 CAL yr. BP or AD 1680–1770 and AD 1800–1940 at the two-sigma level (Figures 1 and 2, Table 4). The second part of the curve, with a probability of 65.1 %, has a greater statistical probability of being correct, suggesting use of this feature between AD 1800 and AD 1940. However, statistical probability doesn't necessarily mean that this date falls within this interval. The historic nature of this feature is also supported by recovery of small amethyst glass fragments. A few fire-cracked rock fragments also were noted in the sample. Uncharred insect chitin and puparia fragments, small uncharred rodent fecal pellets, various uncharred snail shells and snail shell fragments, and uncharred roots/rootlets indicate a moderate level of subsurface disturbance.

The phytolith record is dominated by elongate forms (Figure 3), representing grasses. Moderate quantities of rondels and small quantities of pyramidal rondels and trapezoid forms were noted, representing cool season (festucoid) C_3 grasses. These grasses grow during the cooler months of the year and often in places that trap or hold water. A moderate quantity of chloridoid saddle forms was observed, representing short grasses that thrive in hot, dry conditions. These grasses likely are the most abundant on the landscape. Only a few small bilobate forms were noted that suggest growth of tall warm season (C_4) grasses. These grasses require more moisture than do the short grasses. Buliforms were present, but not particularly abundant. They exhibited surface deterioration suggesting that much of the phytolith, and probably the entire biogenic silica record, was affected by dissolution. A moderate quantity of trichomes, representing plant hairs, also were present. It is likely that most of these originated on grass leaves, although they also might represent other plants. In short, the phytolith record characterizes the local vegetation as including a mixture of short grasses and cool season grasses.

TABLE 1
PROVENIENCE FOR SAMPLES FROM TRINITY-ORACEL PIPELINE #1720,
LA 121520, EDDY COUNTY, NEW MEXICO

Sample No.	Feature	Depth (cmbs)	Provenience/ Description	Analysis
1	4	0-10	Fill including carbon-stained sediments from a fire-cracked rock concentration	Phytolith Macrofloral AMS ^{14}C Date

TABLE 2
MACROFLORAL REMAINS FROM TRINITY-ORACEL PIPELINE #1720,
LA 121520, EDDY COUNTY, NEW MEXICO

Sample No.	Identification	Part	Charred W	Charred F	Uncharred W	Uncharred F	Weights/ Comments
1	Light Fraction Weight						9.374 g
Feat. 4	FLORAL REMAINS:						
0-10	*Celtis*	Seed				1	
cmbs	*Chenopodium*	Seed			2	3	
	Euphorbiaceae	Fruit				35	
	Chamaesyce	Seed			7	13	
	Euphorbia	Seed			19	144*	
	Fabaceae	Leaf			X	X	Moderate
	Prosopis	Seed			1		
	Larrea tridentata	Leaf			X	X	Few
	Larrea tridentata	Seed			9	2	
	Paniceae	Floret			2	1	
	Solanaceae	Seed			6		
	Trianthema portulacastrum	Seed			7	8	
	Roots					X	Few
	Rootlets					X	Numerous
	CHARCOAL/WOOD:						
	Total charcoal $\geq$ 0.5 mm						0.0141 g
	Conifer - vitrified	Charcoal		11			0.0028 g
	Prosopis **	Charcoal		2			0.0036 g
	Unidentified hardwood - small, vitrified	Charcoal		27			0.0077 g
	NON-FLORAL REMAINS:						
	Insect	Chitin				X	Few
	Insect	Puparia				X	Few
	Rock/Sand					X	Few
	Amethyst					X	Few
	FCR					X	Few
	Rodent fecal pellets - small				X	X	Moderate
	Snail shell - depressed				X	X	Few
	Snail shell - oblong				X	X	Moderate

W = Whole
F = Fragment
X = Presence noted in sample
L = Liter
g = grams
mm = millimeters
* = Estimated frequency
**= Submitted for AMS ^{14}C Dating

TABLE 3
INDEX OF MACROFLORAL REMAINS RECOVERED FROM TRINITY-ORACEL PIPELINE #1720, LA 121520, EDDY COUNTY, NEW MEXICO

Scientific Name	Common Name
FLORAL REMAINS:	
Celtis	Hackberry
Chenopodium	Goosefoot, Pigweed
Euphorbiaceae	Spurge family
Chamaesyce (syn. *Euphorbia*)	Sandmat
Euphorbia	Spurge
Fabaceae	Bean family
Larrea tridentata	Creosotebush
Paniceae (includes *Axonopus, Cenchrus*, *Digitaria*, *Echinochloa*, *Panicum*, *Paspalum*, *Pennisetum*, *Setaria*, *Stenotaphrum*, etc.)	A large tribe of the grass family that includes carpetgrass, sandbur, crabgrass, barnyard grass, panicgrass, knotgrass, fountaingrass, bristlegrass, St. Augustine grass, and many others
Prosopis	Mesquite
Solanaceae	Nightshade family
Trianthema portulacastrum	Desert horse purslane
CHARCOAL/WOOD:	
Conifer	Cone-bearing, gymnospermous trees and shrubs, mostly evergreens, including the pine, spruce, fir, juniper, cedar, yew, hemlock, redwood, and cypress
Prosopis	Mesquite
Unidentified hardwood - small	Wood from a broad-leaved flowering tree or shrub, fragments too small for further identification
Unidentified hardwood - vitrified	Wood from a broad-leaved flowering tree or shrub, exhibiting a shiny, glassy appearance due to fusion by heat
NON-FLORAL REMAINS:	
Chitin	A natural polymer found in insect and crustacean exoskeleton
Insect puparium	A rigid outer shell made from tough material that includes chitin (a natural polymer found in insect exoskeleton and crab shells) and hardens from a larva's skin to protect the pupa as it develops into an adult insect
Snail shell - depressed	Snail shell with a depressed (flat) shape where the width is much bigger than the height
Snail shell - oblong	Snail shell with an oblong shape where the height is much bigger than the width

TABLE 4
RADIOCARBON RESULTS FOR SAMPLES FROM TRINITY-ORACEL PIPELINE #1720,
LA 121520, EDDY COUNTY, NEW MEXICO

Sample No.	Sample Identification	AMS ^{14}C Date*	1-sigma Calibrated Date (68.2%)	2-sigma Calibrated Date (95.4%)	$\delta^{13}C$** ($^{o}/_{oo}$)
PRI-14-006-1	*Prosopis* charcoal	123 ± 21 RCYBP	270–210; 150–130; 120–60; 40–20 CAL yr. BP	270–180; 150–10 CAL yr. BP	-25.7
			AD 1680–1740 AD 1800–1820 AD 1830–1890 AD 1910–1930	AD 1680–1770 AD 1800–1940	

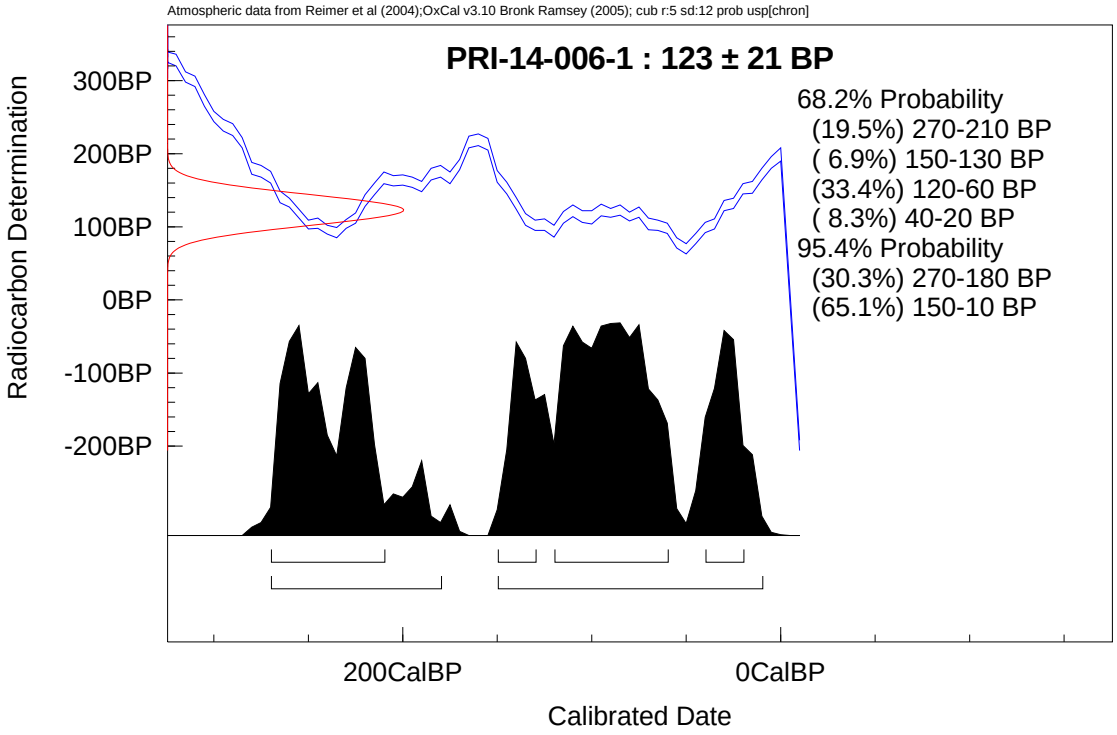
* Reported in radiocarbon years at 1 standard deviation measurement precision (68.2%), corrected for $\delta^{13}C$

** $\delta^{13}C$ values are measured by AMS during the ^{14}C measurement . The AMS-$\delta^{13}C$ values are used for the ^{14}C calculation and should not be used for dietary or paleoenvironmental interpretations.

PaleoResearch Institute
2675 Youngfield Street, Golden, CO 80401
(303) 277-9848 • Fax (303) 462-2700
www.paleoresearch.com

FIGURE 1. PRI RADIOCARBON AGE CALIBRATION.

Laboratory Number: PRI-14-006-1
Sample Identification: *Prosopis* charcoal
Average Lifespan: Variable, depending on species, from 40-200+ years
Conventional AMS ^{14}C Date: 123 ± 21 RCYBP
1-sigma Calibrated Age Range (68.2%): 270–210; 150–130; 120–60; 40–20 CAL yr. BP
2-sigma Calibrated Age Range (95.4%): 270–180; 150–10 CAL yr. BP
$\delta^{13}C$ ($^{o}/_{oo}$): -25.7 (Measured for ^{14}C calculation, not valid for dietary or paleoenvironmental interpretations)



Intercept Statement. For radiocarbon calibration, PRI uses OxCal3.10 (Bronk Ramsey 2005), which is a probability-based method for converting ages in radiocarbon years (RCYBP) into calibrated dates (CAL yr BP). This method is preferred over the intercept-based alternative because instead of providing individual point estimates, it reflects the probability of the date's occurrence within a given range (reflected by the amplitude [height] of the curve). As a result, the probability-based method produces more stable calibrated values than do intercept-based methods (Telford 2004). Ongoing refinements and adjustments to the calibration curve have a greater apparent effect on individual points than on ranges.

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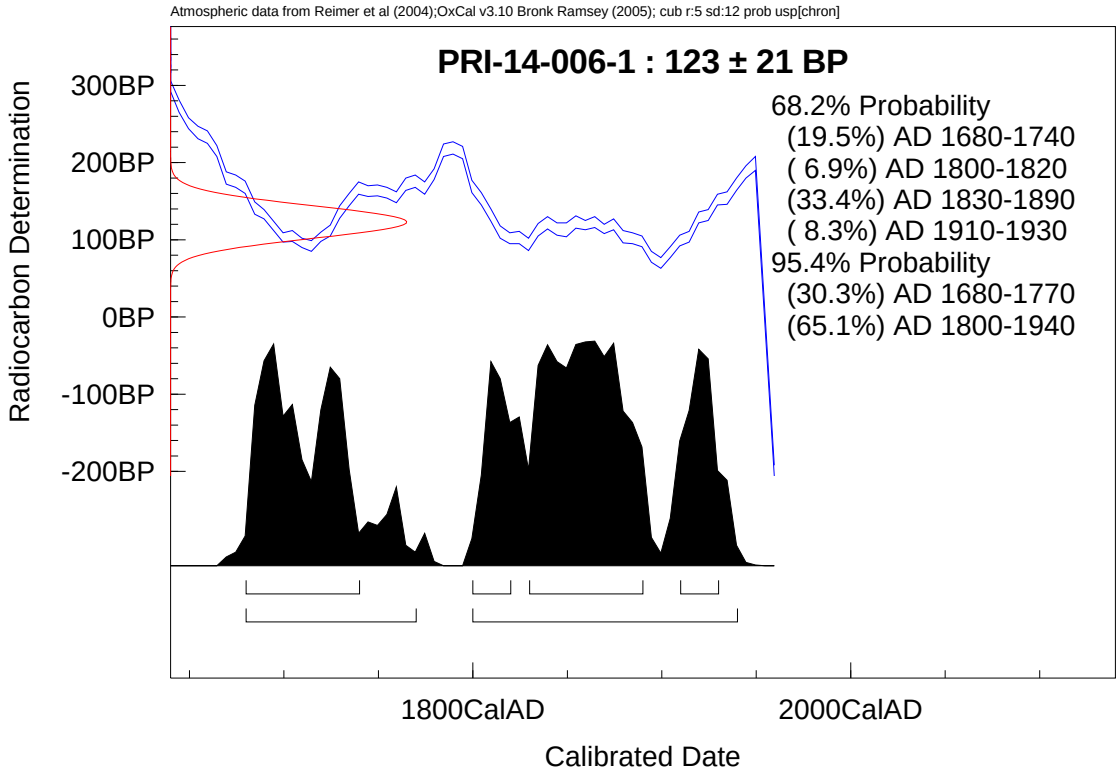
FIGURE 2. PRI RADIOCARBON AGE CALIBRATION.

Laboratory Number: PRI-14-006-1
Sample Identification: *Prosopis* charcoal
Average Lifespan: Variable, depending on species, from 40-200+ years
Conventional AMS ^{14}C Date: 123 ± 21 RCYBP
1-sigma Calibrated Age Range (68.2%): AD 1680–1740; AD 1800–1820; AD 1830–1890; AD 1910–1930
2-sigma Calibrated Age Range (95.4%): AD 1680–1770; AD 1800–1940
$\delta^{13}C$ ($^{o}/_{oo}$): -25.7 (Measured for ^{14}C calculation, not valid for dietary or paleoenvironmental interpretations)



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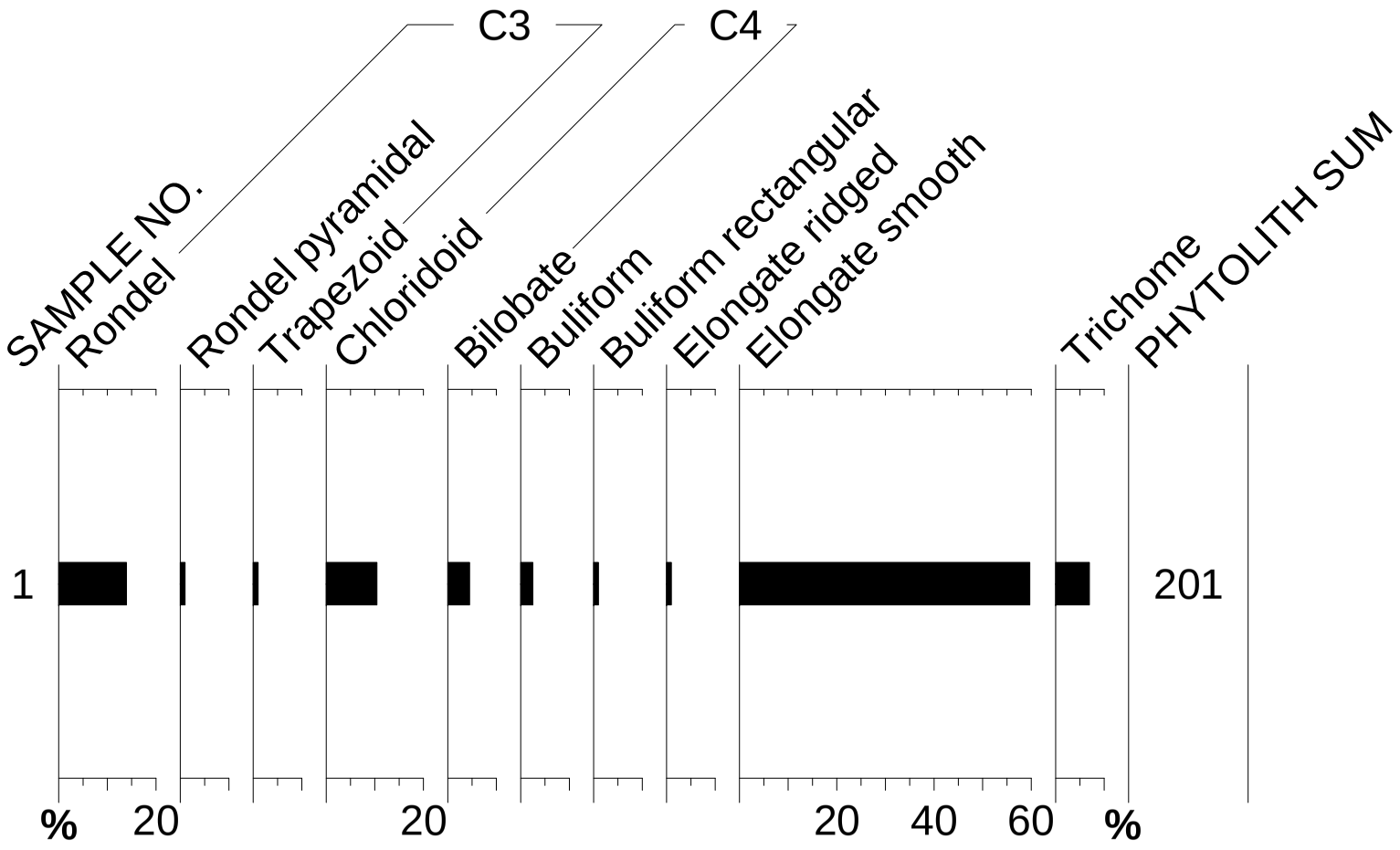

FIGURE 3. PHYTOLITH DIAGRAM, TRINITY-ORACEL PIPELINE #1720, LA 121520.

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