APPENDIX C

Examination of Bulk Soil and Charcoal and AMS Radiocarbon Analysis of Material from the Paradox Valley, Colorado

EXAMINATION OF BULK SOIL AND CHARCOAL AND AMS RADIOCARBON ANALYSIS OF MATERIAL FROM THE PARADOX VALLEY, COLORADO

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INTRODUCTION

Three sediment samples from the Paradox Valley, Colorado, were floated to recover organic fragments suitable for radiocarbon analysis. Two botanic samples also were submitted for identification and radiocarbon dating. These samples were recovered from trenches for a geologic study to determine if the area contains active faults. Botanic components and detrital charcoal were identified, and potentially radiocarbon datable material was separated. A total of four samples were processed for AMS radiocarbon dating.

METHODS

Macrofloral

The sediment samples were floated using a modification of the procedures outlined by Matthews (1979). Each sample was added to approximately 3 gallons of water. The sample was stirred until a strong vortex formed, which was allowed to slow before pouring the light fraction through a 150 micron mesh sieve. Additional water was added and the process repeated until all visible macrofloral 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 fractions were 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 were then examined. Charcoal pieces larger than 1-mm in diameter were broken to expose a fresh cross section and examined under a binocular microscope at a magnification of 70x. The remaining light fraction 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 coarse or heavy fractions also were screened and examined for the presence of botanic remains. Remains from both the light and heavy fractions were recorded as charred and/or uncharred, whole and/or fragments. The individual charcoal and root samples also were broken to expose a fresh cross-section and examined under a binocular microscope at a magnification of 70x.

Macrofloral remains, including charcoal, were identified using manuals (Core, et al. 1976; Martin and Barkley 1961; Panshin and Zeeuw 1980; Petrides and Petrides 1992) and by comparison with modern and archaeological references. The term "seed" is used to represent seeds, achenes, caryopses, and other disseminules. Because charcoal and possibly other botanic remains were to be sent for radiocarbon dating, clean laboratory conditions were used during flotation and identification to avoid contamination. All instruments were washed between samples, and samples were protected from contact with modern charcoal.

Radiocarbon Dating

Wood and charcoal samples submitted for radiocarbon dating are identified and weighed prior to selecting subsamples for pre-treatment. The remainder of each sample, if there is any, is permanently curated at Paleo Research. The subsample selected for pre-treatment is first subjected to hot (at least 110 $^{\circ}$C), 6N hydrochloric acid (HCl), with rinses to neutral between each HCl treatment, until the supernatant is clear. This removes iron compounds and calcium carbonates that would hamper removal of humate compounds later. Next the samples are subjected to 5% potassium hydroxide (KOH) to remove humates. Once again, the samples are rinsed to neutral and re-acidified with pH 2 HCl between each KOH step. This step is repeated until the supernatant is clear, signaling removal of all humates. After humate removal, each sample is made slightly acidic and left that way for the next step. Charcoal samples (but not wood samples) are subjected to a concentrated, hot nitric acid bath, which removes all modern and recent organics. This treatment is not used on unburned or partially burned wood samples because it oxidizes the submitted sample of unknown age.

Each submitted sample is then freeze-dried using a vacuum system, freezing out all moisture at -98 $^{\circ}$C. Each individual sample is combined with cupric oxide (CuO) and elemental silver (Ag^{o}) in a quartz tube, then flame sealed under vacuum.

Standards and laboratory background samples also are treated in the same manner as the wood and charcoal samples of unknown age. A radiocarbon “dead” EUA wood blank from Alaska that is more than 70,000 years old (currently beyond the detection capabilities of AMS) is treated using the same chemical processing as the samples of unknown age in order to calibrate the laboratory correction factor. Standards of known age, such as Two Creeks wood that dates to 11,400 RCYBP and others from the Third International Radiocarbon Intercomparison (TIRI), are also processed simultaneously to establish the laboratory correction factor. Each wood standard is 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 are prepared and sealed in their individual quartz tubes, they are combusted at 820 $^{\circ}$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 ($C0_2$) gas.

Following this last step, all samples of unknown age, the wood standards, and the laboratory backgrounds are sent to the Keck Carbon Cycle AMS Facility at the University of California, Irvine, where the $C0_2$ gas is processed into graphite. The graphite in these samples is then 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 v.3.10. “Bomb dates” are calibrated using CaliBomb. The fraction modern is calibrated using the North Hemisphere Zone 2 radiocarbon data set (NM_zone2.14c dataset)(Hua and Barbetti 2004).

DISCUSSION

The Paradox Valley is located in western Colorado, in the central and southern portions of the state. Cultural, environmental, and geologic studies have been undertaken in this area because of a proposed uranium mill.

Trench 2

Three sediment samples were recovered from Trench 2. Sample STA550 was taken from Unit 4 near the surface (Table 1). This sample contained one small piece of ***Artemisia*** charcoal weighing less than 0.001 g, two small fragments of ***Atriplex*** charcoal weighing less than 0.001 g, and several fragments of hardwood charcoal too small for further identification weighing 0.001 g (Table 2, Table 3). All of these charcoal types were combined to obtain a total charcoal assemblage weighing 0.002 g. This total charcoal was submitted for AMS radiocarbon dating. It yielded a date of 350 ± 15 RCYBP (PRI-08-20-STA550), with a two-sigma calibrated age range of 490-420 and 400-310 CAL yr. BP (Table 4, Figure 1). Sample STA550 also contained several uncharred seeds and rootlets from modern plants, several insect chitin fragments, a few rodent fecal pellets, and a snail shell.

Sample STA560 was collected from Unit 3. An uncharred ***Chenopodium*** fragment, an uncharred ***Monolepis*** seed and eight seed fragments, and a few uncharred rootlets represent modern plants in the area. Small fragments of ***Artemisia*** charcoal weighing 0.001 g and pieces of hardwood charcoal too small for further identification weighing 0.001 g also were noted. An AMS radiocarbon date of 1975 ± 15 RCYBP (PRI-08-20-STA560) was returned for the ***Artemisia*** charcoal from this sample. The two-sigma calibrated age range is 1990-1880 CAL yr. BP (Figure 2). Non-floral remains in this sample include five uncharred bone fragments and a snail shell.

Sample STA540 was recovered from Unit 2 of Trench 2. This sample contained a small fragment of probable ***Artemisia*** charcoal weighing less than 0.001 g and three fragments of hardwood charcoal too small for further identification and weighing less than 0.001 g. This sample was not radiocarbon dated. A few uncharred rootlets represent modern plants. In addition, the sample contained a plastic screw cap lid marked “HW WATER SAVER FAUCET CO.”

Trench 1

Charcoal sample STA326N was recovered from the sandy soil of Trench 1 at a depth of about 7 feet below the surface. This sample consisted of several fragments of ***Juniperus*** charcoal, reflecting juniper wood that burned. These charcoal fragments were not root fragments. The largest fragment of charcoal weighing 0.029 g was processed for AMS radiocarbon analysis. This charcoal yielded a date of 4035 ± 15 RCYBP (PRI-08-20-STA326N), with a two-sigma calibrated age range of 4570-4540 and 4530-4430 CAL yr. BP (Figure 3).

Sample STA327N consists of uncharred root bark fragments noted in the sandy soil near charcoal sample STA326N. A total of 50 root bark fragments weighing 0.982 g were submitted

for AMS radiocarbon dating. A modern “bomb” date with a reported Fraction Modern of 1.3121 ± 0.0019 (PRI-08-20-STA326N) was obtained for these bark fragments, with a two-sigma calibrated age range of January-May 1962, March 1976, December 1976, November 1977-February 1978, and July 1978-August 1979 (Figure 4). This date indicates the presence of “bomb carbon” or elevated quantities of ^{14}C. Atmospheric testing of nuclear weapons resulted in rapidly increasing quantities of ^{14}C in the atmosphere, peaking in 1964-1965. After this, the quantity of ^{14}C has declined in a saw-tooth pattern, as seen in Figure 4. Therefore, a radiocarbon date may intersect the quantity of ^{14}C in two (or more) places on the graph and is usually quite specific, including specifying months of the year for the beginning and ending of individual ranges within the one-sigma and two-sigma calibrations.

SUMMARY AND CONCLUSIONS

Flotation of sediment samples from Trench 2 in the Paradox Valley, Colorado, resulted in recovery of charcoal in sufficient quantities for AMS radiocarbon analysis. Sample STA550 from Unit 4 contained pieces of sagebrush, saltbush, and unidentified hardwood charcoal that were combined to produce a date of 350 ± 15 BP. Sagebrush charcoal in sample STA560 from Unit 3 yielded a date of 1975 ± 15 BP. Juniper charcoal and unburned root bark were found near one another at a depth of about 7 feet below the surface in Trench 1. The juniper charcoal yielded a date of 4035 ± 15 BP. The unburned root bark revealed the presence of “bomb carbon”, indicating that the roots were alive anywhere from 1962-1979. The presence of “bomb carbon” is certain information that the unburned roots were alive after World War II when atomic bombs were deployed. The introduction of the extra carbon as a result of exploding atomic bombs both underground and in the atmosphere made dating within the “post bomb” era extremely accurate. Because of the fact that carbon isotopes increased very rapidly after this time, any samples containing “bomb carbon” can be dated very precisely to either the time of rising carbon (in the 1950s) or the time of declining carbon (in the 1990s).

TABLE 1
PROVENIENCE DATA FOR SAMPLES FROM THE PARADOX VALLEY, COLORADO

Sample No.	Trench No.	Unit No.	Provenience/ Description	Analysis
STA 550	2	4	Bulk sediment	Macrofloral AMS ^{14}C Date
STA 560	2	3	Subsidence Material	Macrofloral AMS ^{14}C Date
STA 540	2	2	Bulk sediment, Upper clay	Macrofloral
STA 326N	1		Charcoal in sandy soil	Charcoal ID AMS ^{14}C Date
STA 327N	1		Uncharred root	AMS ^{14}C Date

TABLE 2 (Continued)

TABLE 2
MACROFLORAL REMAINS FROM THE PARADOX VALLEY, COLORADO

Sample No.	Identification	Part	Charred W	Charred F	Uncharred W	Uncharred F	Weights/ Comments
STA550	Liters Floated						1.75 L
Trench 2	Light Fraction Weight						5.86 g
Unit 4	FLORAL REMAINS:						
	Asteraceae	Seed				1	
	Cheno-am	Perisperm				1	
	Atriplex	Seed			1	10	
	Chenopodium	Seed			2	5	
	Chenopodium - reticulate	Seed			1	2	
	Descurainia	Seed				1	
	Rootlets					X	Moderate
	CHARCOAL/WOOD:						
	Artemisia**	Charcoal		1			<0.001 g
	Atriplex**	Charcoal		2			<0.001 g
	Unidentified hardwood - small	Charcoal		21			0.001 g
	NON-FLORAL REMAINS:						
	Insect	Chitin				30	
	Rock/Gravel					X	Moderate
	Rodent fecal pellet					X	Few
	Snail shell					1	
STA560	Liters Floated						2.25 L
Trench 2	Light Fraction Weight						6.87 g
Unit 43	FLORAL REMAINS:						
	Chenopodium - reticulate	Seed				1	
	Monolepis	Seed			1	8	
	Rootlets					X	Few
	CHARCOAL/WOOD:						
	Artemisia**	Charcoal		6			0.001 g
	Unidentified hardwood - small	Charcoal		13			0.001 g
	NON-FLORAL REMAINS:						
	Bone					5	
	Rock/Gravel					X	Few
	Snail shell				1		

TABLE 2 (Continued)

Sample No.	Identification	Part	Charred W	Charred F	Uncharred W	Uncharred F	Weights/ Comments
STA540	Liters Floated						1.50 L
Trench 2	Light Fraction Weight						3.24 g
Unit 2	FLORAL REMAINS:						
	Rootlets					X	Few
	CHARCOAL/WOOD:						
	cf. ***Artemisia***	Charcoal		1			<0.001 g
	Unidentified hardwood - small	Charcoal		3			<0.001 g
	NON-FLORAL REMAINS:						
	Plastic cap - screw lid				1		
	Rock/Gravel					X	Moderate
STA326N	Volume Water-screened						<0.10 L
Trench 1	Water-screened Sample Weight						18.94 g
	FLORAL REMAINS:						
	Rootlets					X	Few
	CHARCOAL/WOOD:						
	Juniperus**	Charcoal		18			0.14 g
	NON-FLORAL REMAINS:						
	Sand					X	Moderate
STA327N	Volume Water-screened						<0.10 L
Trench 1	Water-screened Sample Weight						7.21 g
	FLORAL REMAINS:						
	Root bark**					X	Moderate
	Rootlets					X	Few
	NON-FLORAL REMAINS:						
	Sand					X	Few

W = Whole
F = Fragment
X = Presence noted in sample
g = grams
**= Submitted for AMS Radiocarbon Dating

TABLE 2 (Continued)

TABLE 3
INDEX OF MACROFLORAL REMAINS RECOVERED FROM THE PARADOX VALLEY, COLORADO

Scientific Name	Common Name
FLORAL REMAINS:	
Asteraceae	Sunflower family
Cheno-am	Includes goosefoot and amaranth families
Atriplex	Saltbush, Shadscale
Chenopodium	Goosefoot, Pigweed
Monolepis	Povertyweed
Descurainia	Tansy mustard, Flixweed
CHARCOAL/WOOD:	
Artemisia	Sagebrush
Atriplex	Saltbush, Shadscale
Juniperus	Juniper
Unidentified hardwood - small	Wood from a broad-leaved flowering tree or shrub, fragments too small for further identification

TABLE 4
RADIOCARBON RESULTS FOR SAMPLES FROM THE PARADOX VALLEY, COLORADO

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-08-20-STA550	***Artemisia***, ***Atriplex***, and unidentified hardwood charcoal	350 ± 15 RCYBP	470-420, 380-320 CAL yr. BP	490-420, 400-310 CAL yr. BP	-23.9
PRI-08-20-STA560	***Artemisia*** charcoal	1975 ± 15 RCYBP	1950-1895 CAL yr. BP	1990-1880 CAL yr. BP	-25.4
PRI-08-20-STA326N	***Juniperus*** charcoal	4035 ± 15 RCYBP	4530-4510, 4485-4445 CAL yr. BP	4570-4540, 4530-4430 CAL yr. BP	-18.6
PRI-08-20-STA327N Bomb Date	Unburned root bark	1.3121 ± 0.0019 Fraction Modern	January-March 1962December 1979September 1978-January 1979 CAL yr. BP	January -May 1962 March 1976 December 1976 November 1977-February 1978 July 1978-August 1979	-15.4

* 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.

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