Radiocarbon Dating Rock Paintings at Lost Canyon West Shelter (41CX1019) on the Twistflower Ranch, TX

Archaeological Research & Education Center

Karen L. Steelman & Vicky Roberts
Shumla Archaeological Research & Education Center
P.O. Box 627, Comstock, Texas, 78837 • 432-292-4848 •
info@shumla.org • www.shumla.org

Introduction

This research project is a collaboration with archaeologist Mike Quigg and landowner Mike McCloskey to radiocarbon date the rock paintings within Lost Canyon West Shelter (41CX1019) on the Twistflower Ranch in Crockett County, Texas. The ability to place rock art in a chronological context allows an inventory of images to be studied together with excavated cultural remains from a given archaeological time period and geographical area. This incorporation of rock art studies alongside other archaeological specialties is crucial for developing a synergistic approach to studying past cultures.

Figure 1. View into Lost Canyon West Shelter (41CX1019) looking west. The pictographs (not visible) are located on the shelter wall and ceiling.

Site Description

41CX1019 is a multi-component site comprised of an upland lithic scatter, and a series of three rockshelters located just below the limestone canyon rim: one with pictographs (Figure 1); one with a spring; and one with an animal den. Prickly pear, acacia, and native grasses are present. The top most portion of the site consists of a large lithic scatter while the lowest portion contains two shelters. One of the lower shelters contains a permanent spring with ~12 deeply incised groove marks and three large mortars positioned within and just outside the shelter. The other lower shelter has very large, coarse-sized rocks and spalls; there were no cultural materials observed when investigated.

Located near the top of the bluff and accessible from the uplands, the main rockshelter, Lost Canyon West Shelter, contains traces of red and black pigment (Figure 2). This pictograph rockshelter has a bedrock floor with no deposits and a low, slanted limestone ceiling. On Panel 1 within the main shelter, there are approximately 21 enigmatic figures and 24 areas of remnant paint. There are fine line black paintings mostly panel right and lower down on the shelter wall. The shelter also contains dark red paint, as well as a less intense red paint. Most of the red paintings are remnants that can no longer be discerned. The fine line black paintings consist of four concentric circle or spiral motifs/grouping, horizontal zig zag lines, and at least three lines with V-shapes along its length, as if depicting a plant stalk or a feather. The top left spiral is clearly a spiral with a beginning and an ending. The other motifs in the grouping are less clear and may be concentric circles. Some of the paintings have scratch marks along the length of their lines as post-painting modification by prehistoric peoples (Figure 3). The fine line execution of the black paintings is reminiscent of Red Linear, but there are no characteristic anthropomorphs or zoomorphs or hunting scenes to make us suspect that this unknown style is related. It the execution of the painting and their relative size that they have in common, but the subject of the art is different. Some of the black fine line painting was superimposed with red remnants, but the painting order has not been determined. As this is an unknown style, determining the age of the art will help place this unknown style in context with other surrounding rock art sites.

Radiocarbon Dating Rock Art

The archaeological chemistry laboratory at Shumla Archaeological Research & Education Center employs a custom-built plasma oxidation apparatus to convert organic material to carbon dioxide for accelerator mass spectrometry (AMS) radiocarbon dating (Steelman and Rowe 2012). This technique is particularly amenable to dating rock paintings, as it extracts only the organic fraction and leaves the inorganic matrix (Russ et al. 1990). Glow discharges are produced by radio frequency (RF) capacitive coupling with two external copper electrodes on either end of a glass sample chamber (Figure 4). A plasma is an electrically excited gas composed of neutral atoms, both negative and positive molecular and atomic ions, and electrons. Neon signs and fluorescent lights are plasmas commonly used by society. Electrons gain kinetic energy from an oscillating electric field, while the temperatures of the gas components are increased by elastic collisions between the electrons and the gas. Electrons are thermally isolated from the gas components by their very large mass differences. Temperatures of the plasma gas thus can remain near ambient temperatures at the same time the electrons are sufficiently energetic to break molecular bonds. The active species in the plasma phase allow reactions that normally occur only at high temperatures to proceed at low temperatures. Oxygen plasmas convert organic matter to carbon dioxide and water, which we collect by freezing the products with liquid nitrogen for AMS radiocarbon dating.

Figure 2. Some of the best preserved rock art at Lost Canyon West Shelter consists of fine line black paintings of possible spirals and feather or plant like images.

Figure 3. Post-painting modification in the form of incised grooves in an area of red painting.

Figure 4. Rock art sample in chamber during argon plasma exposure.

Methods

Sample Collection

While each dated motif was chosen carefully for the archaeological information that would be obtained, the individual samples were collected from already fragile areas. These were locations that we would have eventually lost due to weathering. Thus, sampling was done to minimize the impact of collection on these images.

One black and one red pigmented paint samples were collected by K. Steelman and V. Roberts for analysis. Sample #1 is from a red remnant painting that has no discernable shape. Sample #2 is from a fine line, black, miniature painting. In addition, two samples (#1b & #2b) from adjacent unpainted rock were also collected as backgrounds to investigate contamination levels of organic material in the rock substrate. Photographs of sampling locations were taken before and after collection (Figures 5-16). Individual sterile surgical scalpels were used to collect each paint sample from a surface area on the order of 1 to 3 cm^2. All paint and background samples were stored in folded aluminum foil squares (Figure 17) and placed in labeled plastic bags until commencement of laboratory analysis. To avoid contamination, samples were manipulated only with aluminum foil or scalpel blades.

Figure 5. Sample #1 is from a dark red paint remnant that was radiocarbon dated to 1950±35 years BP. This photograph was taken before the paint sample was collected.

Figure 6. Location after removing sample #1 from the shelter wall.

Figure 7. Context photograph showing the location for sample #1.

Figure 8. A control sample of unpainted rock was collected adjacent to paint sample #1. This photograph was taken before background sample#1b was collected.

Figure 9. Location after removing sample #1b from the shelter wall.

Figure 10. Context photograph showing the location for sample #1b.

Figure 11. Sample #2 is from black paint just to the left of the fine line painting at the site (obscured by the right hand). Sample #2 of black paint was radiocarbon dated to 1965±35 years BP.

Figure 12. Location after removing sample #2 from the shelter wall.

Figure 13. Context photograph showing the location for sample #2.

Figure 14. A control sample of unpainted rock was collected directly below the location of paint sample #2. This photograph was taken before background sample#2b was collected.

Figure 15. Location after removing sample #2b from the shelter wall.

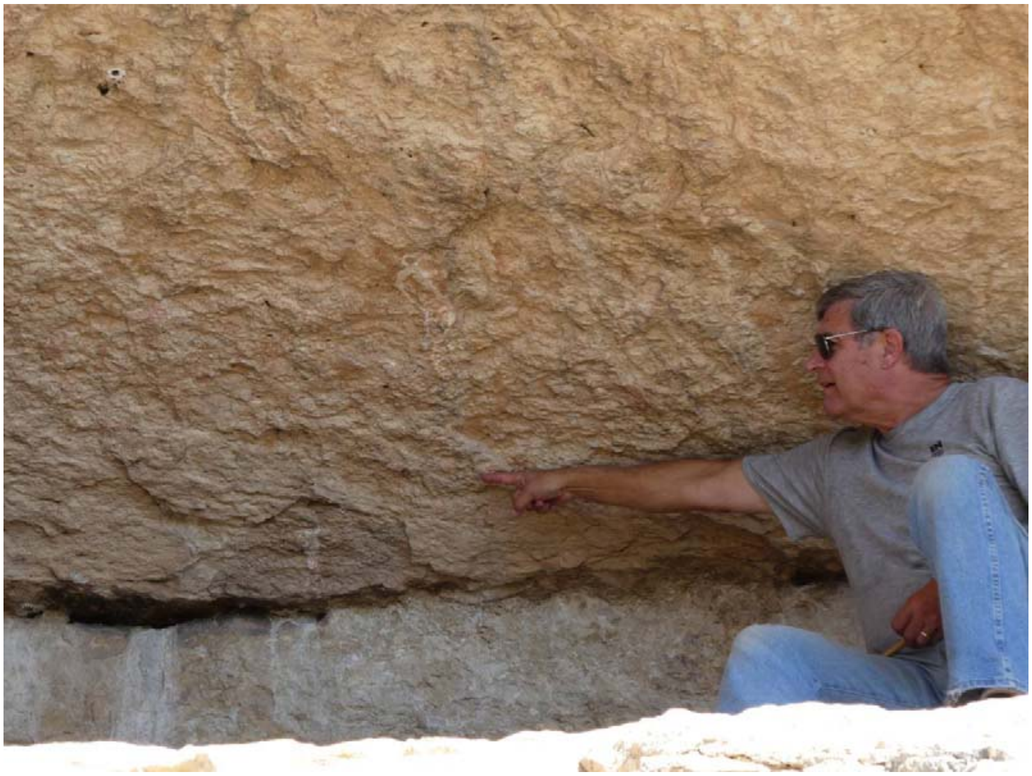

Figure 16. Context photograph showing the location for sample #2b.

Figure 17. Collecting a sample with a sterile scalpel blade and aluminum foil square.

Chemical Pretreatment

The samples arrived at the Shumla laboratory as rock flakes, as the collected samples were from potential spalls. Therefore, we used individual sterile surgical scalpel blades to remove the outer paint layer as a powder. The resultant powder was then examined with a stereoscope at X40 magnification to remove any visible contaminants such as plant fibers, rootlets, or spider webs with micro-tweezers. No visible contaminants were observed for these samples. We then weighed the samples into sterile, plastic centrifuge tubes.

Typically, both paint samples and unpainted rock backgrounds are subjected to chemical pretreatment to remove humic contamination. Humic acids, naturally present in soil samples and derived from the decay of organic matter, appear brownish-orange in a basic solution. From our experience, the presence of humic acids is minimal for paint samples collected on rock substrates, but we continue this practice as a precaution.

For the paint layer and background samples, we added 3 mL of dilute (1 Molar) sodium hydroxide solution to the centrifuged tubes and placed them in an ultrasonic water bath at 50±5°C for one hour in order to remove any potential humic material. After base treatment, the samples were centrifuged for 10 minutes. The liquid above all but one sample was colorless and transparent, indicating that no humics were present. After decanting the basic liquid above the solid samples, 3 mL of de-ionized water was added to each tube. The sample tubes were placed in an ultrasonic water bath at 50±5°C for another hour. After the tubes were centrifuged, the liquid was decanted and the solid samples were stored in de-ionized water until ready for filtration.

We used vacuum filtration with a water aspirator to collect the solid samples onto quartz-fiber filters that had been previously baked at 500°C in a muffle furnace. A trap was used to prevent potential backflow of water and contaminants coming into contact with the samples. After filtration was complete, filters were dried in an oven at 110°C, then wrapped in foil and stored in a desiccator.

Plasma Oxidation

The Shumla laboratory employed a custom-built plasma oxidation apparatus to convert organic material in paint and background samples to carbon dioxide for accelerator mass spectrometry (AMS) radiocarbon dating. The apparatus, kept under vacuum at a pressure of $<1x10^{-6}$ torr, utilized research-grade oxygen (99.998%) and ultra-high purity argon (99.999%) gases to minimize contamination. The apparatus sample chamber was cleaned by igniting successive oxygen plasma reactions at 1 torr oxygen gas and 100 or 150 watts radio frequency power for one hour each until ≤0.3 μg carbon is remaining. These cleaning oxygen plasma reactions removed any organic material on the inside of the chamber introduced by previous samples or modern contamination from handling.

Next, we loaded a sample into the chamber and evacuated the system overnight using a turbomolecular pump with a scroll fore pump to a pressure of <1x10^{-6} torr. Successive argon plasma discharges, at 1 torr and 40 Watts radio frequency power for one hour, were ignited to remove adsorbed gasses by surface ablation of the sample. Argon was used because it is an inert gas and will not react with organic material in a sample. Once a pressure reading of <1 millitorr was obtained, it indicated that all adsorbed gasses were removed (≤0.3 μg carbon).

Finally, the sample was oxidized with an oxygen plasma at a pressure of 1 torr and 100 Watts radio frequency power. The organic material in the sample was converted into carbon dioxide and water during the one-hour exposure. The product carbon dioxide was flame-sealed into a glass tube cooled to liquid nitrogen temperature (-194°C), after the water was removed with a dry ice/ethanol slurry (-58°C). If sufficient carbon was obtained, the collected glass tube was sent to the Center for Accelerator Mass Spectrometry (CAMS) at Lawrence Livermore National Laboratory for graphitization and radiocarbon measurement.

Results & Discussion

Age results are shown in Table 1 and Figures 18 & 19. The $\delta^{13}C$ values were assumed to be -25‰, as no split of the carbon dioxide was sampled for stable carbon isotope measurement. Carbon levels in unpainted rock backgrounds were negligible suggesting that extracted carbon is from the paint samples alone and contamination from the rock substrate is not significant (Table 1). During plasma oxidation, the color of the paint samples persisted, indicating that the paints were made with mineral pigments instead of an organic material such as charcoal for the black color. The radiocarbon dates for both paint samples are statistically indistinguishable and pass a χ^2-test indicating that they are coeval, with a weighted average of 1958±25 years BP calibrated to 40 cal BC – 120 cal AD at 2 sigma (95.4% probability) using the R_Combine function of the OxCal computer program version 4.3.2 (Bronk Ramsey 2009, 2017) with IntCal13 curve data from Reimer et al. (2013) (Figure 20).

Conclusions

Two radiocarbon dates on pictographs at Lost Canyon West Shelter (41CX1019) have statistically indistinguishable ages. A dark red remnant is 1950±35 years BP and a black fine line painting is 1965±35 years BP. A weighted average of these two results calibrates to 40 cal BC – 120 cal AD at 2σ probability. This unknown painting style can now be compared to other contemporaneous artifacts from the area to better understand the prehistoric peoples of Crockett County.

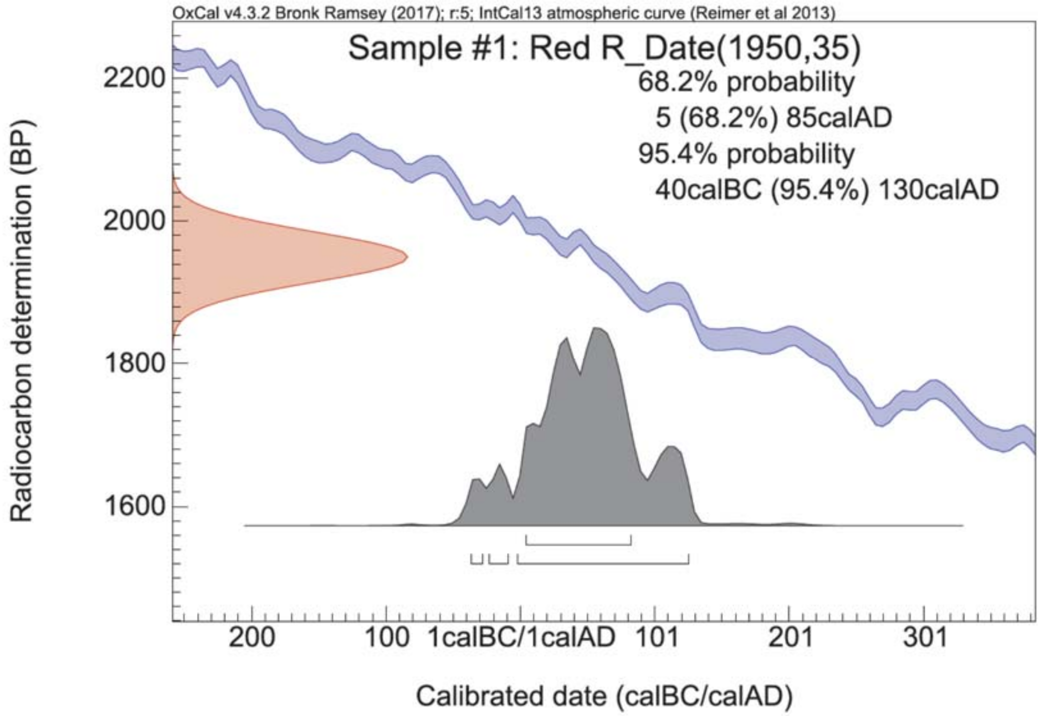

Figure 18. OxCal calibration for Sample #1 of red paint.

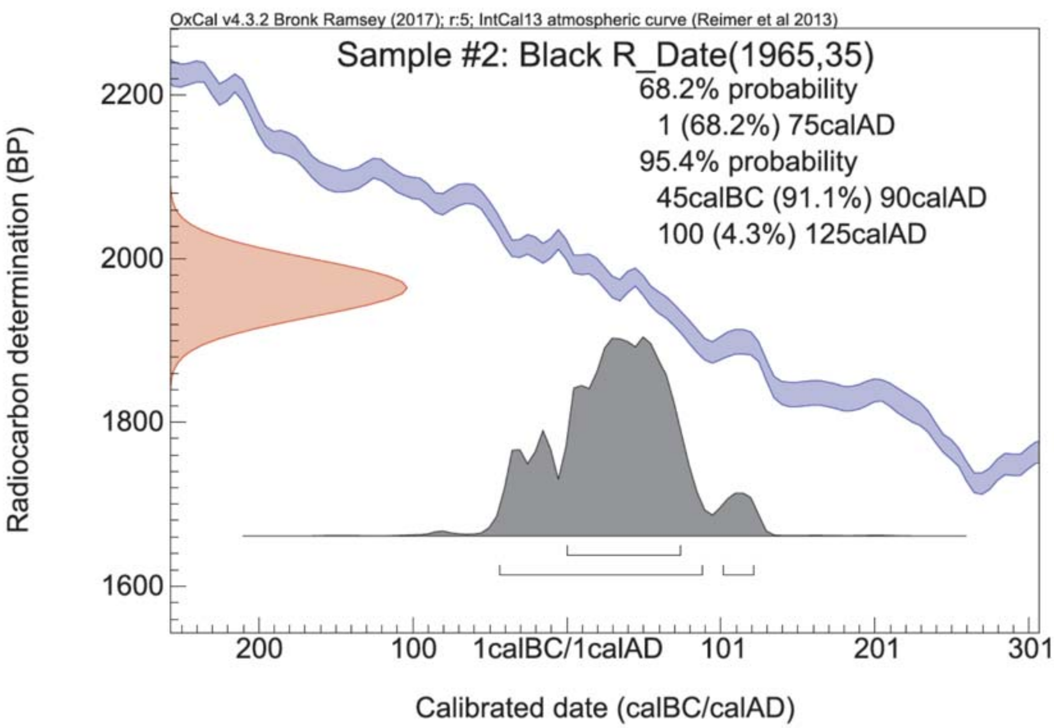

Figure 19. OxCal calibration for Sample #2 of black paint.

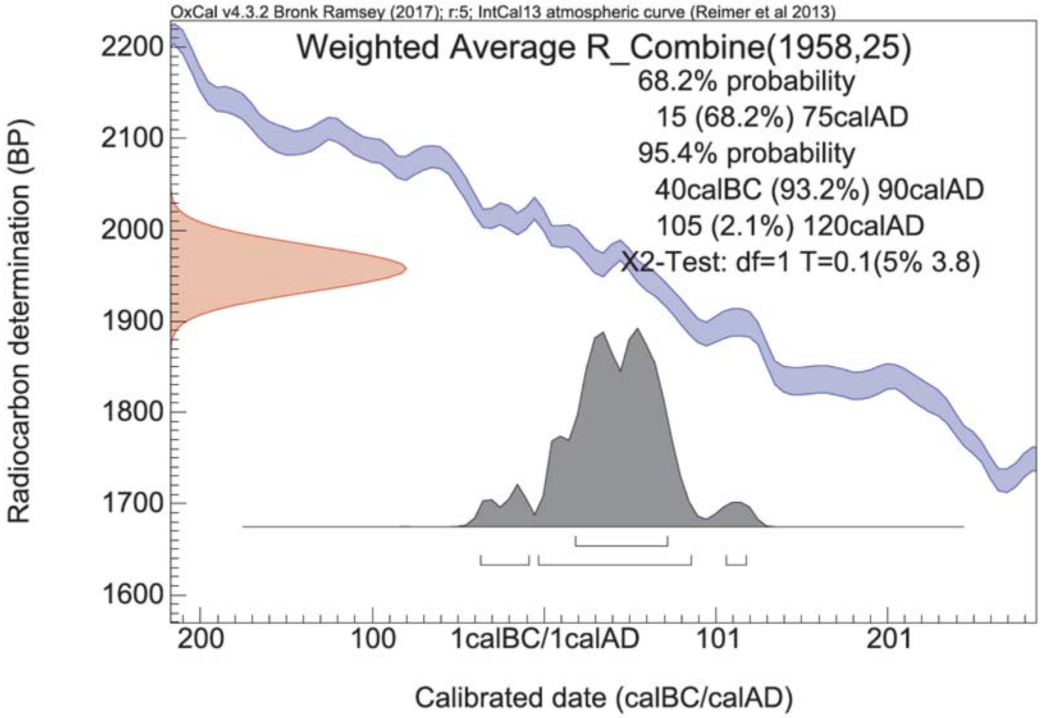

Figure 20. Weighted average for the two statistically indistinguishable radiocarbon dates obtained at Lost Canyon West Shelter.

Table 1. Radiocarbon Results for 41CX1019

Sample	Description	Mass (g)	Extracted C (µg)	µg/g ratio	Shumla CO_2 ID	CAMS ID	^{14}C Date (BP)	Calibrated Range (2σ, 95.4%)
1	dark red	2.80	147	50	18	179736	1950±35	40 cal BC – 130 cal AD
1b	background	2.48	3	1	--			
2	black	0.31	240	770	17	180153	1965±35	45 cal BC – 90 cal AD (91.1%) 100 cal AD – 125 cal AD (4.3%)
2b	background	1.36	3	2	--			
Weighted average							1958±25	40 cal BC – 90 cal AD (93.2%) 105 cal AD – 120 cal AD (2.1%)

Acknowledgements

We thank the Mike McCloskey and his late son, Ted McCloskey, for hosting us during a documentation and sampling trip at their Twistflower Ranch. We are honored that you shared your beautiful ranch with us. We also thank archaeologist, Mike Quigg, for his guidance and interest in this project.

References Cited

Bronk Ramsey, C. 2009 Bayesian analysis of radiocarbon dates. 2009 *Radiocarbon* 51:337-360.

Bronk Ramsey, C. 2018 On-line *OxCal version 4.3.2* available at *https://c14.arch.ox.ac.uk/oxcal.html*

Reimer, P.J., E. Bard, A. Bayliss, J.W. Beck, P.G. Blackwell, C. Bronk Ramsey, P.M. Grootes, T.P. Guilderson, H. Haflidason, I. Hajdas, C. Hatte, T.J. Heaton, D.L. Hoffmann, A.G. Hogg, K.A. Hughen, K.F. Kaiser, B. Kromer, S.W. Manning, M. Niu, R.W. Reimer, D.A. Richards, E.M. Scott, J.R. Southon, R.A. Staff, C.S.M. Turney & J. van der Plicht. 2013. IntCal13 and Marine13 Radiocarbon Age Calibration Curves 0-50,000 Years cal BP. *Radiocarbon* 55: 1869-1887.

Russ, J.; Hyman, M.; Shafer, H. J.; Rowe, M. W. 1990 Radiocarbon dating of prehistoric rock paintings by selective oxidation of organic carbon. *Nature* 348: 710-711.

Steelman, K. L. and Rowe, M. W., 2012, Radiocarbon dating of rock paintings: incorporating pictographs into the archaeological record, in *A Companion to Rock Art* (eds. J. McDonald and P. Veth), pp.565-582, Blackwell Companions to Anthropology series, Wiley-Blackwell Publishing, Oxford, UK.