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Stable Isotope and Radiocarbon Analyses of a Black Deposit Associated with Pictographs at Little Lost River Cave, Idaho

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A glossy, black deposit covers much of the ceiling and walls of Little Lost River Cave No. 1, Idaho. This site is of particular interest because of the red, orange, and yellow pictographs underlying the coating. Carbon and nitrogen stable isotope analysis has allowed us to better understand the nature and origin of the deposit. With a $\delta^{13}C$ value of $-20{\cdot}1$‰, $\delta^{15}N$ value of $+14$‰, and a C/N ratio of 5·6 after removal of inorganic carbon from the sample matrix, the deposit appears to have been derived from animal tissue, not plant. Plasma chemical extraction of carbon from the organic material in another sample of the deposit, followed by accelerator mass spectrometric analysis, yielded a minimum uncalibrated radiocarbon age of 2990 ± 50 BP for the paintings. This preliminary evidence suggests that the deposit may be a cooking residue.

Keywords: STABLE CARBON- AND NITROGEN-ISOTOPE ANALYSIS, PLASMA CO_2 EXTRACTION, AMS RADIOCARBON DATING, ORGANIC MATERIAL, PICTOGRAPHS, SHOSHONE, FREMONT, ARCHAIC.

Introduction

A glossy, black coating covers many red, orange, and yellow rock paintings (pictographs) at Little Lost River Cave No. 1 in southeastern Idaho. The deposit is a shiny, malleable coating that is located only in the back chamber of the cave. The coating overlying the pictographs makes them visible only when viewed from certain angles with artificial light. Artificial light is necessary because the shelter is deep enough that sunlight inadequately illuminates the area under question. Black deposits found in caves and on rock shelter walls are common worldwide and include a variety of types and origins. Black deposits on limestone and marble buildings and monuments (Amoroso & Fassina, 1983; Urzì, Krumbein & Warschei, 1992) and specifically in caves (Smith, Bouchard & Lorblanchet, 1999) have been shown to contain several different materials, including manganese minerals (Moore, 1981; Peck, 1987), coloration from black fungi or bacteria (Diakumaku *et al.*, 1995; Gorbushina *et al.*, 1993; Groth *et al.*, 1999), oxalate crusts (Edwards *et al.*, 1992; Russ *et al.*, 1996), decomposing plant materials or humates, tar, and bat or pack-rat guano (Hill, 1982). From our preliminary analyses, the black deposit at Little Lost River Cave No. 1 does not appear to be any of these types of previously studied materials. Instead, it appears to be

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Figure 1. Photograph showing the entrance of Little Lost River Cave No. 1 located near Howe, Idaho.

similar to a decomposed olive residue identified as a black deposit at Cueva del Encajero, Spain (Saiz-Jimenez & Hermosin, 1999). Though similar, we believe that the black deposit at Little Lost River Cave No. 1 is of animal origin, perhaps a cooking residue.

A sample of the black deposit was effervescent when placed in acid (suggesting the presence of carbonates) and a fine black residue remained insoluble in acid and in water. An electron probe microanalysis (EPMA) with both energy- and wavelength-dispersive X-ray capabilities strongly suggests that the matrix of the deposit contains organic material (Steelman *et al.*, in press, 2001). The black coating consists of carbonates and iron oxide/hydroxide dust clasts uniformly suspended in a carbon-rich supporting matrix. Also from EPMA, substantial amounts of chlorine and boron were observed both throughout the black deposit matrix and a 1–2 µm accretion on the surface of "clean" rock substrate. Stable isotope analysis here shows that some of the carbon in the sample is organic, confirming a conclusion reached much earlier (Fichter *et al.*, 1955).

Given that the alternative source of carbon in the black deposit in Little Lost River Cave No. 1 is organic, and assuming that it was formed from contemporaneous carbon, this material can be used to establish a relevant minimum age for those paintings covered by it. While radiocarbon dating is a possibility, incorporation of carbon from old wood or old charcoal sources into the deposit is an issue. To obtain a meaningful radiocarbon date, the deposit must either be the result of anthropogenic activity (either deliberate or inadvertent) or a geological event that occurred in the Holocene. If the deposit were an application of geologically derived "dead" hydrocarbons (e.g. tar or pitch) or modern carbon, a radiocarbon age would not relate to the time of painting activity at the site.

In this current study, we conducted carbon and nitrogen elemental and isotopic analyses and measured the radiocarbon activity of the black deposit in Little Lost River Cave No. 1. We have measured the amount of organic carbon in a sample of the deposit. Stable isotope data suggest an animal origin for this organic material, while the radiocarbon date establishes the deposit as an Archaic archaeological event.

Site Description

Little Lost River Cave No. 1 (Site 10BT1) is located in Butte County, Idaho, within the Little Lost River drainage about 6 miles from the southeastern tip of the Lemhi Range, one of a series of three fault block ranges in east-central Idaho. The cave, estimated at about 5650 ft above sea level, is a solution cavity in dolomitic limestone. The inorganic carbonates that make up the limestone were probably formed during the Carboniferous Age ($\sim$300 million years old) and contain only "dead" carbon (i.e., no ^{14}C remaining). The southeast opening is at the base of a limestone outcrop and at the top of a gentle colluvial slope (Figure 1). With an opening about 15 m across and between 2 and 3 m tall at the highest point, the cave is roughly 17 m in length towards the northwest (Butler, 1981*a*). The cave has two chambers separated by a low ceiling, but the black deposit occurs only in the back chamber, which opens up to a height of about 2 m (Figure 2).

Little Lost River Cave No. 1 was first studied archaeologically in 1954 with an exploratory excavation conducted by Idaho State College (Fichter *et al.*, 1955; summarized by Butler, 1981*a*). Before its excavation, local artifact hunters vandalized the site and then later brought the cave to the attention of the college. Further excavations were carried out in 1990

Figure 2. Photograph of the cave interior showing the black deposit coating the ceiling and walls of the back chamber.

by archaeologists from the University of Alberta, Canada (Gruhn & Bryan, 1990). The 1990 report concluded from point types (Humboldt Concave-based, Northern Side-notched, Elko Corner-notched, and stemmed indented base points) that the main occupation phase at the site was during the Middle Prehistoric Period (4000 BC–AD 400). Both excavation reports mention pictographs in the cave and a black glossy coating that covers much of the walls and ceiling of the cave. A partial chemical analysis in the 1954 report identified the substance to be organic in nature, containing carbon, hydrogen, and oxygen.

The pictographs in the cave were "rediscovered" in 1999 during a BLM cost share project with Archaeographics to record all rock art sites in the Black Canyon Wilderness Assessment Area. Since then, the cave has been mapped to show the location of the pictographs. However, the reflective glare of the black coating made photography and drawing of the individual panels in the back chamber a challenging experience (Figure 3). It was only after careful scrutiny of each image under a variety of light sources that the superimposition of the pictographs in relation to the deposit could be determined. Photo electronic imaging techniques applied to scanned negatives and slides of the pictographs helped further clarify many of the motifs. A cross-section shows the location of a pictograph pigment layer underneath the black deposit (Figure 4). Thus, the age of the deposit corresponds to a minimum date for the pictographs.

Located in the Black Canyon Wilderness Area, the site rests in the territorial domain of the Lemhi Shoshone and the Shoshone-Bannock tribes. Although the Shoshone believe they have always lived in this area, multiple hypotheses purport to explain human and cultural migration and influence in the Snake River Plain from Archaic hunter-gathers to a Fremont presence to Shoshone (Swanson, 1972; Butler, 1981*a*, *b*, 1986; Adovasio, Andrews & Fowler, 1982; Holmer, 1994). Considering these varied hypotheses, a significant contribution to the cultural history of the Snake River Plain can be made if the material in the black deposit yields an accurate minimum radiocarbon age for the pictographs and if the stylistic and ethnographic evidence can identify a cultural influence for the images in Little Lost River Cave No. 1.

The Pictographs

It is difficult to discuss pictographs in Little Lost River Cave No. 1 without considering the relationship of the images to the shiny black deposit. Pictographs appear on three types of rock environments: (1) the majority, located in the back chamber, are covered by the black deposit; (2) those on top of a white (calcite) deposit; and (3) a few on "clean" rock substrate. All images are either red, orange, or yellow ochre with a few in white crayon, probably a beige clay or light yellow ochre. Definitive color assignments are difficult for the pictographs under the coating because it is not known how the coating may affect viewing the original pigment. This is especially true for the white-appearing pigment that is only seen through the black coating. A majority of the pictographs look as if they were drawn with a crayon or pigment stick because of their stippled appearance. A few solid colored figures appear to have been painted because the pigment is evenly dispersed over the rock in finger-width lines.

For recording purposes (Merrell, 2001), the cave was divided into six sections determined by the placement of pictograph groupings along the walls and ceiling. Groups of motifs that filled a natural segment of rock were labelled as panels within a section. Only section

(A)

(B)

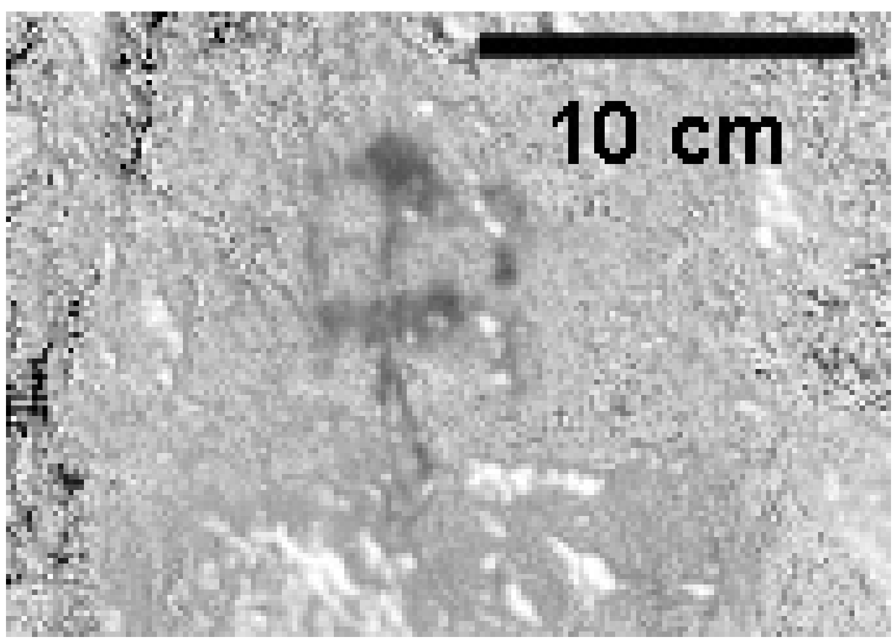

Figure 3. (A) Tracing of pictograph covered by the black deposit made from an enhanced photograph and (B) enhanced photograph of the same image.

one, which contained a yellow horned zoomorph and an anthropomorph, occurs in the front chamber of the cave and is free of the black coating. Also in the front chamber, four red-orange isolated motifs were painted on segments of rock suspended from the ceiling; two were stick anthropomorphs, one was a shield bearer, and one was a single finger-width line of pigment. The variety of motifs from sections two through six in the rear chamber includes zoomorphs, humanoids, spears, rakes, a complex fine line grid, and a large number of geometric abstract shapes, many of which are superimposed. When viewed as a whole, many of the abstract designs, grids and rakes resemble entoptic patterns seen during trance-induced states as discussed by Lewis-Williams & Dowson (1988) and Whitley (2000).

There are two other motifs that may relate to shamanism. The first is the identification of a “Mugua” or power sphere (Lowie, 1909) This term is used to describe a solid round ball that appears directly above

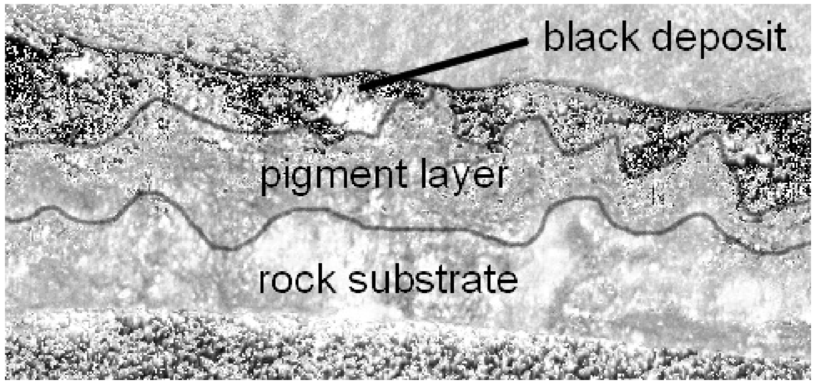

Figure 4. Cross-section showing a pigment layer between the rock substrate and black deposit. The cross-section microphotograph taken with an optical stereoscope was enhanced before converting to grayscale. An outline was added around the pigment layer for emphasis. The microphotograph width is approximately 3 mm.

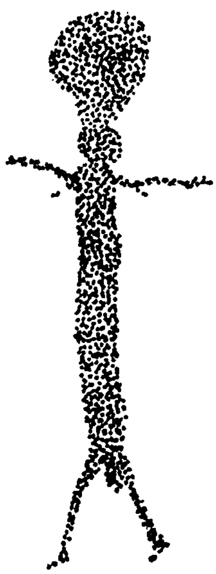

Figure 5. Red-orange anthropomorph with ''mugua'' above head. The image is 16 cm tall.

the head of a human figure (Figure 5). Use of this symbol may indicate the spirit leaving the body, rising above the head, as a spiritual connection is made with a greater power. This can occur during a ''death'' experience when the body may not actually die, but experience an altered state of consciousness. Alternatively, a small ball on top of the head is described as a ''shaman's knot'' because a shaman often wore his hair tied up on his head in such a fashion (M. Pavesic, pers. comm., 2001). Both cases would help identify that figure as a shaman. The second motif looks like one body rising up out of a lower human figure (Figure 6). In this case there appears to be an upper torso with a double set of arms and head. This figure may symbolize the spirit leaving the body during a trance-induced state. Both of these motif types are found at other sites considered very sacred to the Shoshone-Bannock people.

Recent investigations of pictograph sites in the southern Lemhi Mountains, including the Little Lost River Cave No. 1, by Merrell (1999, 2000, 2001) indicate an affiliation with the ancient Shoshone. At the Big Spring pictograph site, located nearby in a western canyon of the Lemhi range, Shoshone-Bannock people are actively seeking protection for an area they consider sacred to their people (Yupe, 2001). Many motifs found at this site resemble images also found beneath the black deposit in Little Lost River Cave No. 1 and at other sites in the Black Canyon Wilderness Area. Stylistic relationships between motifs at these sites and claims of the Shoshone-Bannock must be considered. With stylistic and ethnographic evidence pointing toward a Shoshone cultural affiliation to the images found in the cave, a minimum radiocarbon date of the pictographs will help establish the cultural history of the Snake River Plain.

Experimental Methods

During September 2000, we sampled the black deposit for study, including carbon and nitrogen stable isotope analysis and radiocarbon dating. We removed approximately 1 gram of the coating that was peeling or flaking from the surface. We used new, sterile surgical scalpels and wore rubber gloves. The sample, including incorporated calcium carbonate and accretionary mineral matter, was placed on aluminium foil, wrapped, and stored in resealable plastic bags. The black material was then taken to Texas A&M University for analysis. The surface of each flake was examined with an optical microscope to ensure that no visible extraneous materials (roots, etc.) were included in the sample to be studied; none was found.

Carbon and nitrogen elemental and isotopic analyses

The sample (black material and incorporated accretionary minerals) was dried at 60°C in a forced-air oven, pulverized to a fine powder with a mortar and

Figure 6. Yellow ochre body torso rising from lower body painted on a white precipitate wash. The anthropomorph is 17 cm tall.

pestle, mixed thoroughly to ensure homogeneity, and an aliquot was weighed (± 1 μg) into a tin foil capsule using a microbalance (ATI Cahn Model C-44, Boston, MA). The sample was then analysed for $\delta^{13}C$, $\delta^{15}N$, %C, and %N using a Carlo Erba EA-1108 elemental analyser interfaced with a Delta Plus (Finnigan MAT, Bremen, Germany) isotope ratio mass spectrometer operating in continuous flow mode. A second aliquot was weighed (± 1 μg) into a silver foil capsule, treated in-capsule with 10% HCl to volatilize carbonate-carbon, dried, and analysed for organic carbon concentration (%) and $\delta^{13}C$ of organic carbon as above (Nieuwenhuize, Mass & Middelburg, 1994). Previous studies showed that acid treatment has no significant effect on the $\delta^{13}C$ and %C of organic matter (Midwood & Boutton, 1998). $\delta^{13}C$ values are expressed relative to the V-PDB standard (Coplen, 1995), while $\delta^{15}N$ values are expressed relative to AIR (Mariotti, 1983). Precision (± 1 S.D.) was 0·06‰ for $\delta^{13}C$, 0·12‰ for $\delta^{15}N$, 0·16% for %C, and 0·01% for %N.

Radiocarbon dating

On a second sample of this black deposit, we utilized the plasma-chemical extraction method in order to separate the organic carbon in the deposit from "dead" inorganic carbon. This technique has been used to separate organic carbon from rock art pigment samples for radiocarbon dating and has been described in detail elsewhere (e.g., Russ *et al.*, 1990; Rowe, 2001). Ultra-high purity bottled argon and oxygen (99·999%) gases were used for all plasmas. A rotary pump and a turbo molecular pump maintained vacuum conditions ($\sim 10^{-4}$ and $\sim 10^{-7}$ torr, respectively). First, low-temperature oxygen plasmas were used to pre-clean the reaction chamber; these were repeated until $\leq$0·001 mg carbon, as CO_2, was released. Once the chamber was clean, approximately 15 flakes of the deposit ($\sim$3–5 mm in diameter) were placed into the chamber with their outer, shiny surface exposed. This black deposit sample received no prior chemical pre-treatment. Samples were introduced into the chamber via a stainless-steel, copper-gasketed, flange port under a flow of argon to prevent atmospheric CO_2, aerosols or organic particles from entering the system.

After the chamber was resealed and the sample degassed somewhat under vacuum, five low temperature (<150°C) argon plasmas for one hour each were used to desorb CO_2 molecules from the sample and chamber walls by inelastic collisions of non-reactive,

Table 1. Results of carbon and nitrogen elemental and isotopic analyses

Treatment	$\delta^{13}C_{V\text{-}PDB}$	%C	$\delta^{15}N_{AIR}$	%N	C/N
Pulverized only	−1·9‰	12·1%	+14·3‰	0·47%	25·7
Pulverized+Acidified	−20·1‰	2·8%	+14·9‰	0·50%	5·6

but energetic, argon species. The amount of adsorbed CO_2 and water on the sample was thus diminished. However, because the sample contains volatile compounds, presumably organic, we were not able to reduce the vacuum to correspond to <0·001 mg carbon equivalent as is usual in our procedure. Nonetheless, after conducting five argon plasmas, we expect that adsorbed CO_2 was essentially removed and virtually all gaseous products frozen with liquid nitrogen (from the last argon plasma) were from volatile organic compounds in the sample.

Next, low-temperature (≤150°C), low-pressure (~1 torr) oxygen plasmas oxidized organic components of the sample to CO_2. We collected and saved the CO_2 extract from the first hour-long oxygen plasma reaction. A second oxygen plasma produced 0·15 mg of carbon as carbon dioxide during 37 min of plasma exposure. Decomposition of inorganic carbon present (dolomitic limestone rock and calcite/calcium oxalate accretions) was prevented by running the plasmas at low-temperature. The oxidizing plasmas react only with organic carbon present in the samples, leaving substrate rock and accretionary carbonates and oxalates intact (Russ, Hyman & Rowe, 1992; Chaffee, Hyman & Rowe, 1994). Carbon dioxide from the sample was flame-sealed into a glass tube cooled to liquid nitrogen temperature (−194°C), after water had been frozen out with a dry-ice/ethanol slurry (−58°C). The CO_2 from the second oxygen plasma was sent for radiocarbon analysis at the Center for Accelerator Mass Spectrometry at the Lawrence Livermore National Laboratory (LLNL-CAMS). It was necessary to utilize an AMS measurement due to the small sample size.

Results and Discussion

Carbon and nitrogen isotopic and elemental analyses

Prior to treatment with acid, the pulverized whole sample (black material and incorporated accretionary minerals) consisted of 12·1% total carbon and 0·47% total nitrogen (Table 1). Based on the $\delta^{13}C$ value of −1·9‰, it was clear that most of the carbon in the whole sample was inorganic, probably from $CaCO_3$ (which is 12% carbon). This was expected because the black material was deposited on dolomitic limestone. Thus, most of the total carbon is of geological origin.

After carbonate-carbon was volatilized from the sample by acidification, elemental and isotopic analyses of the black residue revealed that it consisted of 2·8% carbon and had an organic $\delta^{13}C$ of −20·1‰ (Table 1). The $\delta^{13}C$ of the residue is well within the range of values reported previously for proteinaceous organic carbon, but higher (less negative) than that typically found in soils and terrestrial C3 plants (Boutton, 1991). Similar $\delta^{13}C$ values have been reported for lichens from rock walls in the Lower Pecos River region of southwest Texas (Russ *et al.*, 1999). However, we saw no evidence of lichen in these samples, either visually or in EPMA backscattered electron images. Furthermore, low light intensities characteristic of this cave environment precluded the presence of lichens.

Both $\delta^{15}N$ and %N of the sample were unaffected by acid treatment (Table 1). The $\delta^{15}N$ value we measured is relatively enriched, and greater than values usually associated with terrestrial plants and soils, approximately −8 to +8‰ (Handley *et al.*, 1999). However, because there is a stepwise enrichment of ^{15}N of +3·5‰ with every trophic transfer along a food chain (Minagawa & Wada, 1984), it is plausible that the value of +14‰ for this cave deposit reflects an animal origin. Some herbivores, numerous carnivore species, and humans, have $\delta^{15}N$ values of similar magnitude (Ambrose, 1986, 1991; Grocke, Bocherens & Mariotti, 1997).

The C/N ratio of the cave sample was 5·6 after acid treatment (Table 1). Since C/N ratios of plants are 20–50 and soils are generally 10–12 (Stevenson & Cole, 1999), this organic deposit is not likely derived from those sources. However, C/N ratios of animal tissues are generally near 5–7, again suggesting that this material is of animal origin. Given that the deposit is of animal origin, the $\delta^{13}C$ value of −20·1‰ may be adjusted to correct for ^{13}C enrichment. There is a 3–5‰ enrichment of ^{13}C for bone collagen (Bender, Baerris & Steventon, 1981; DeNiro & Epstein, 1978; van der Merwe, 1982). Therefore, organic material in the deposit most likely originated from animals that were exposed to a diet with a $\delta^{13}C$ value of approximately −24‰, resembling C3 plant material. The precise average value for C3 plants is −27‰ and the deposit may be even more enriched in ^{13}C due to a C4 or CAM plant presence in the animals' diet.

One possible explanation for the formation of the deposit is that animal organics were volatilized by cooking fires, and condensed onto the relatively cool walls of the cave. In fact, previous studies showed that C/N ratios of cooked meat residues on cookware were 4–6 (Hastorf & DeNiro, 1985), similar to this deposit in Little Lost River Cave No. 1. This organic residue

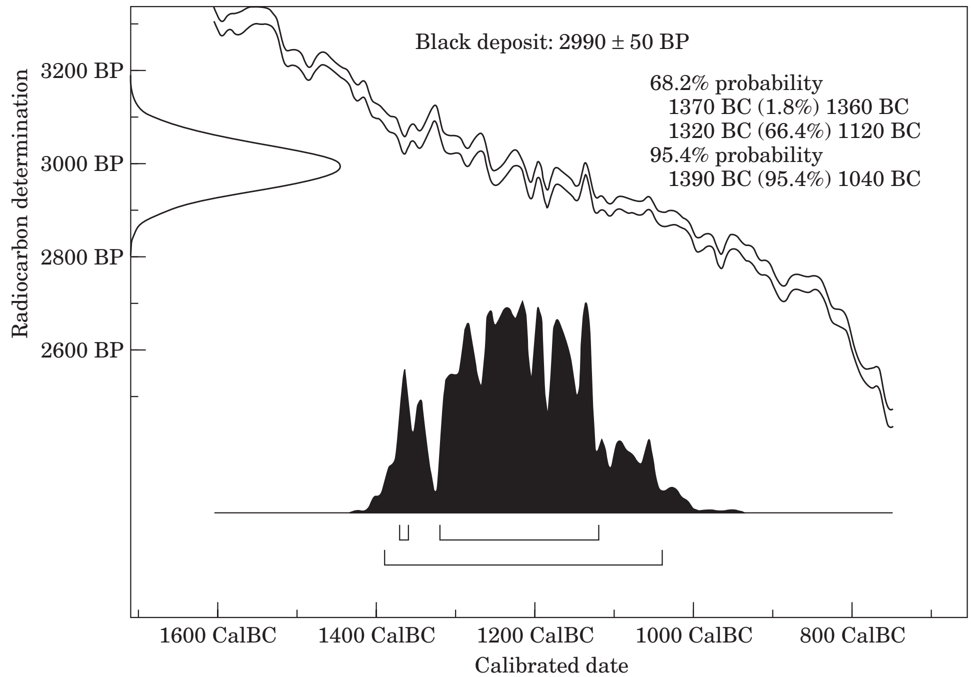

Figure 7. Calibration curve for the radiocarbon date of the black deposit using OxCal v3.5 (Ramsey, 2000; Stuiver *et al.*, 1998).

could have then been trapped by deposition of chloride and borate salts.

Radiocarbon date

The radiocarbon date of 2990 ± 50 BP measured at LLNL-CAMS (#72240) corrected for $\delta^{13}C$ of −20·1‰ corresponds to a calibrated radiocarbon age range of 1390 BC to 1040 BC with 95·4% probability (2 S.D.) shown in Figure 7. The antiquity of this black deposit makes the sample of interest to archaeologists. Issues surrounding charcoal radiocarbon dates such as old wood and old charcoal are not pertinent in this case because of the animal source of the carbon in the deposit.

Conclusions

Carbon and nitrogen isotopic and elemental analyses suggest that the organic material in the black deposit on the ceiling and walls of Little Lost River Cave No. 1, Idaho, was derived from an animal source, rather than from plant material. In further support of our conclusions, Fitcher *et al.* (1955) and Gruhn & Bryan (1990) both mention an irregular charcoal lens as well as the presence of charred and splintered bones of antelope, deer, sheep, and/or bison during excavation. In light of the animal source of the black deposit, a more detailed faunal analysis and dating of charcoal may offer a correlation with our results. Only when a better notion of the deposit formation is known will we be able to state with certainty whether the deposit is a natural one, or whether it was somehow caused by ancient Indigenous people of the region. The radiocarbon date obtained, 2990 ± 50 BP, places the formation of the deposit as a distinctly prehistoric event and this minimum date for the paintings indicates that the pictographs are older than previously thought. This preliminary evidence suggests that the deposit is a cooking residue from "barbecuing" antelope, deer, sheep, and/or bison in the cave.

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