

THE RELATIONSHIP OF MINERALOGICAL DATA TO PALEONTOLOGICAL QUESTIONS: A CASE STUDY FROM CATHEDRAL CAVE, WHITE PINE COUNTY, NEVADA

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Abstract This study describes the mineralogy of sediment samples taken from a paleontological excavation in Cathedral Cave in eastern Nevada. Sediment samples were composed mostly of calcite and gypsum, and a few samples contained minor amounts of quartz and halite. A discrete cemented layer was present throughout portions of the excavated area. The primary mineral constituents of the cemented layer were nitratine (i.e., nitratite and soda niter) and halite, although a sample near the top of the layer was composed of Mg-calcite. Spherical pockets of powdery white gypsum were found intermittently at lower depths. The deposition of the nitratine and the gypsum pockets is likely the result of a leached guano layer. However, the presence of soluble nitratine and soluble halite may be a proxy for very arid conditions at the age of deposition. Mineralogical data can provide an independent source for addressing questions related to a variety of topics (e.g., paleoenvironments and depositional context), and we suggest that paleontologists who conduct excavations in caves may want to incorporate mineralogical analyses as part of their research program.

INTRODUCTION

Paleontological studies of fossiliferous cave deposits are common, and in most of these research projects, attempts are made to identify changes in features of the excavated sedimentary sequences (e.g., Barnosky et al., 2004). Those changes (e.g., grain-size, color, etc.) are often interpreted as distinct stratigraphic and/or chronologic layers. However, few of these types of studies incorporate detailed observations relating to mineralogical changes through the excavated stratigraphic sequences.

Chemical and mineralogical inputs into cave systems can be distinct from surface settings (Gillieson, 1996). As a result, data concerning these inputs may improve understanding of the unique depositional context of fossils collected from a particular cave setting and can potentially provide an independent source of data for other types of research questions (e.g., paleoenvironmental questions). The primary objective of this research project was to examine the mineralogy of an excavated section of Cathedral Cave, Nevada in order to understand better the depositional context of fossils excavated from the site. In this paper we describe the mineralogical content of excavated sediments from Cathedral Cave based on transmitted polarized light microscopy of thin-sections, X-ray diffraction (XRD), and scanning electron microscope (SEM) examination. We report a rare mineralogical record for the state of Nevada and discuss possible causes for some of the unique mineralogical characteristics observed. Finally, we offer suggestions for improving similar types of studies.

SITE DESCRIPTION

Cathedral Cave is located in the northern Snake Range in eastern White Pine County, Nevada within the east-central portion of the hydrographic Great Basin (sensu Grayson, 1993; Fig. 1). The cave entrance is situated on a north-facing slope within middle Cambrian rocks that crop out at the mouth of Smith Creek Canyon. Middle Cambrian formations in the Snake Range include (from oldest to youngest) the Pole Canyon Limestone, Lincoln Peak Formation, and Johns Walsh Limestone (Hose and Blake, 1976). Cathedral Cave is situated within one of these formations, but presently it is not known which.

The cave has two north-facing entrances and only three major rooms (Fig. 1). The paleontological excavations that provided the samples for this project were carried out in Room 2 by a joint team from The University of Texas at Austin and Northern Arizona University in June 2003. Age estimates for the excavated materials are currently being revised, but likely pre-date the terminal Pleistocene (Jass, 2005).

EXCAVATION METHODS

Sediments excavated from Cathedral Cave were collected using standard paleontological techniques. Horizon-

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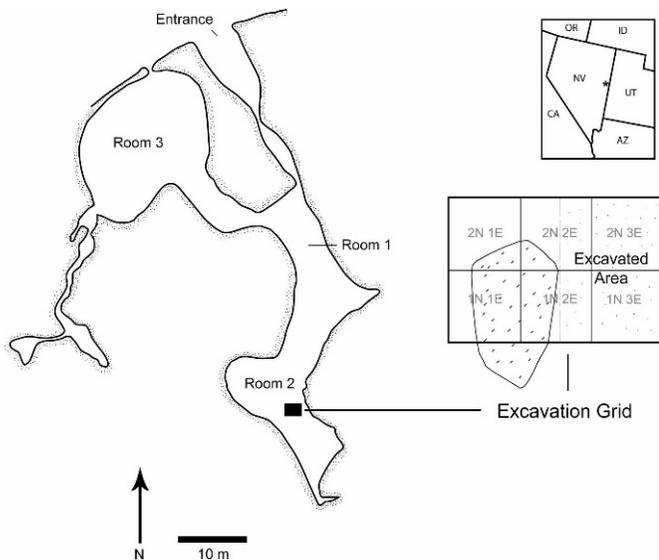


Figure 1. Map of Cathedral Cave showing geographic locations (inset) and the area included in the 2003 excavation. Sediment samples for this study come from the area demarcated in 1N 2E, 2N 2E, 1N 3E, and 2N 3E.

tal control was maintained by devising a 1 m × 1 m grid system. Vertical control was maintained by excavating in arbitrary 5-cm intervals except in instances where natural sedimentary breaks were evident (e.g. grain size, color, cemented versus non-cemented). In such cases, natural sedimentary levels were collected as distinct samples. The majority of excavated sediments were dry-screened on site using nested 6.35 mm and 0.7 mm mesh box screens in order to reduce excess matrix.

SITE STRATIGRAPHY AND SAMPLE SELECTION

Sediments removed during the excavation at Cathedral Cave consisted primarily of an amalgamation of vertebrate bone and unconsolidated silty clay sediments. Grain-size for the majority of sediments (excluding bone) was estimated to be fine or smaller and no notable changes in grain-size were observed through the excavated sequence. Minor color variations were noted (see Fig. 2), but were not deemed significant enough to warrant the excavation of distinct layers. Minor color differences observed in cross-section were not readily evident in plan view as the excavation proceeded.

During the excavation, three discrete sedimentary features were observed. A layer of cemented matrix and bone was encountered in four of the excavation grids between depths of 42 centimeters below datum (cmbd) and 57 cmbd (Fig. 2). The upper and lower boundaries of this level were easily identified as the excavation proceeded and the level was collected as a discrete sample. This layer was not horizontally continuous throughout the entire excavated area and did not extend into the northernmost portions of excavation. Sediments below the cemented

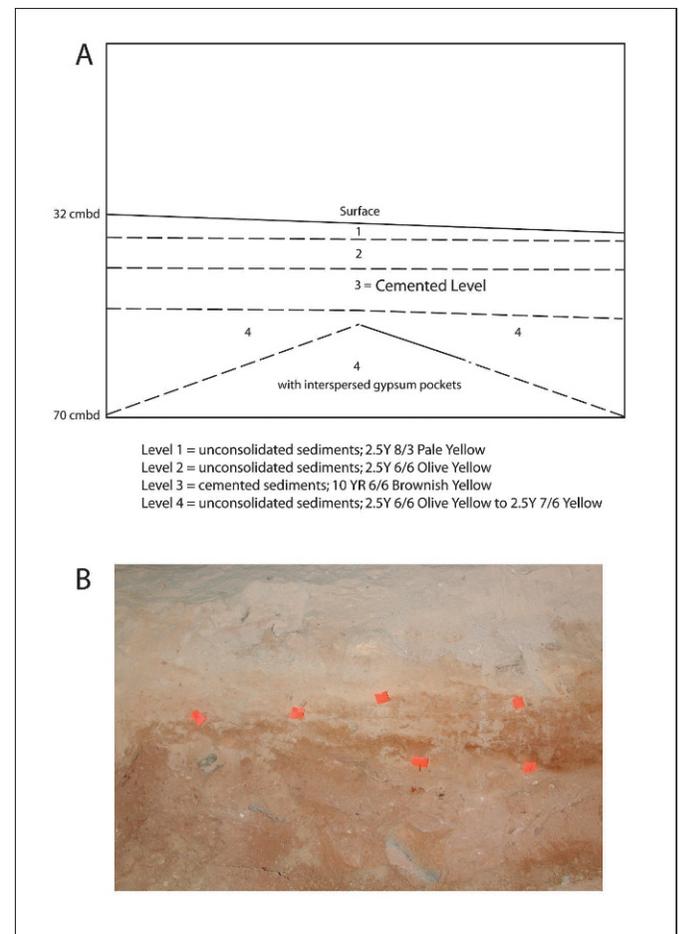


Figure 2. Sedimentary features discussed in text. A. Generalized cross-section from the north wall of excavation square 1N3E indicating overall sedimentary character of deposit. Color changes are noted using descriptions from a Munsell color chart. B. Photograph of the south wall of the excavation showing the cemented level. The top and bottom of the cemented level is bracketed by orange tape.

layer were similar to those occurring above the cemented layer in that they were composed primarily of a homogeneous, unconsolidated matrix. A second observable stratigraphic feature was the presence of discrete pockets of fine-grained, spherical, powder-like, white sediments that occurred sporadically throughout the excavation at 53 cmbd and below (Fig. 2). These pockets occurred below the cemented layer. Finally, several collected bones were encrusted with mineral deposits. These occurred above, within, and below 5 cm of the cemented level.

Samples of the spherical white sediments, samples of bones with encrusting minerals, and some samples from the cemented level were collected separately on site and were used in the analyses presented here. Other samples from the top of the cemented level or within the cemented level were subjected to the dry-screening process but remained consolidated. Samples of unconsolidated sediments were

selected from those materials that were dry-screened during fieldwork at the site. A complete summary of samples and their provenience is presented in Appendix 1.

LABORATORY METHODS

Light microscopy of thin-sections, X-Ray diffraction (XRD) analysis, and scanning electron microscope (SEM) examination were used to identify minerals in excavated sediments from Cathedral Cave. A total of 14 thin sections were impregnated with blue epoxy and cut to 30 μm thickness. Two thin sections from the cemented level were made two months after the other sections were prepared. Special care was taken to prepare these two sections, because XRD data suggested these samples contained nitratine and/or halite. These minerals are both highly soluble, so these thin sections were cut using a dry saw (Nesse, 2000).

In our XRD analyses, we used a Siemens D500 diffractometer fitted with a Cu X-ray tube. Samples were ground to a fine powder and scanned from 5° to 65° 2θ . Operating conditions for the tube were 45 kV accelerating voltage at 30 milliamps. XRD data were collected using the automation controller 'DataScan' by MDI, Inc. XRD patterns were analyzed using the matching routines of JADE 6+ by MDI, Inc., and using the 'Minerals 2003 ICDD' database.

SEM analyses were conducted using a model JEOL T330A at an accelerating voltage of 20 kV. Samples were gold coated using the Denton Desk II Gold Sputter Coater. Gold was sputtered at 50 millitorr in a vacuum at 45 milliamps for 30 seconds. All samples except for the bones exhibiting evidence of secondary mineral deposition were gold coated for an additional 20 seconds at 50 millitorr in order to obtain better control of sample charging.

RESULTS AND DISCUSSION

SEDIMENTS ABOVE CEMENTED LEVEL

Light microscopy

Light microscopy of the two thin-sections made from above the cemented level showed the samples to be composed mostly of angular to sub-angular particles of unidentified grains. Bone was recognizable in both thin sections. Angular calcite grains were evident and commonly contained micro-fractures. These micro-fractures may be artifacts of thin-section preparation. Some conical grains evident in thin-section are likely derived from speleothems.

In both thin-sections, many grains were coated with unidentified clay-sized particles. The texture of these grains may be the result of precipitation of microcrystalline materials on the grains after deposition but before excavation. Alternatively, clay-sized particles may have accumulated around the edges of the grains as they were being agitated during the dry-screening. Another explanation for the coating of grains is that during transportation of the sediments (in plastic Ziploc bags) from the field to the laboratory there was

an increase in humidity within the bags that resulted in the accumulation of mud around these grains.

XRD analysis

XRD-analyses from sediments from above the cemented layer confirmed the presence of calcite. One sample shows a peak consistent with aragonite (35–40 cmbd). In two samples, XRD-analysis showed pronounced peaks consistent with halite. Thin sections from these levels were made using water, so it is possible that any evident halite grains were dissolved away during preparation. These same samples also showed peaks consistent with quartz, but quartz was not identified in thin section. Quartz may be a constituent of the microcrystalline materials observed throughout the thin section.

SEM analysis

SEM-analyses of sediments above the cemented level were uninformative. No distinct mineralogical structures were noted.

TOP OF CEMENTED LEVEL

The cemented level was first encountered between 40–45 cmbd. Numerous samples were taken from sediment bags at this horizon. Because the mineralogy of the excavated section becomes unique at this horizon, we draw a distinction between the top of the cemented level and the cemented level. One of our samples from 40–45 cmbd was loosely-cemented and broke easily in hand sample. This sample was distinct from more well-cemented materials at lower depths in the cemented level. Other sediments from the top of the cemented level at 40–45 cmbd were unconsolidated and loose.

Light microscopy

The loosely-cemented sample from 40–45 cmbd had particles of bone that were observable by the naked eye and in thin section. The color and texture of that sample resembled bat guano, so there was focus towards discerning phosphate minerals. The index of refraction of material from that sample was less than 1.5 (phosphate minerals have indices of refraction larger than 1.5), so it is certain that phosphate minerals were not dominant constituents. Phosphate may be present, but amorphous.

XRD analysis

XRD-analyses of the loosely-cemented sample from 40–45 cmbd showed it to be dominated by Mg-calcite. Comparisons of the XRD pattern from this sample to huntite, a common magnesium-rich carbonate ($\text{CaMg}_3(\text{CO}_3)_4$) known to occur as an evaporite in caves (Hill and Forti, 1997), were inconclusive.

XRD-analysis of a sample of unconsolidated sediments from 40–45 cmbd showed peaks consistent with nitratine, halite, calcite, and gypsum. Nitratine (NaNO_3), also called nitratite (Hutchinson, 1950) and soda niter (Gaines et al.,

1997), is isostructural with calcite (Hill and Forti, 1997), and exhibits many similar optical properties to calcite (such as high birefringence and rhombohedral cleavage).

SEM analysis

No SEM-analyses of sediments from the top of the cemented level were conducted.

CEMENTED LEVEL

Light microscopy

Thin section analyses of the stratigraphically lower, cemented-level samples showed the dominant phase occurring as porous or spongy bladed aggregate. These samples were well-cemented as compared to the samples from the top of the cemented level (40–45 cmbd). A predominance of nitratine in XRD analyses suggests that nitratine is most likely the cementing mineral. Calcite and nitratine are essentially indistinguishable in light microscopy.

XRD analysis

Three XRD-analyses of samples taken from cemented level were dominated by nitratine. Smaller amounts of calcite, gypsum, and bone also were present. Two bone samples exhibiting secondary mineral deposition were taken from the cemented level and analyzed using XRD. XRD-analysis also showed distinct peaks indicative of nitratine with smaller amounts of halite.

Three additional tests were performed to confirm the presence of nitratine in Cathedral Cave. Comparisons of XRD data with nitratine from The University of Texas mineral collection (Figs. 3a and 3b), dissolution and re-precipitation of the mineral, and a taste-test following Gaines et al. (1997) supported the identification of nitratine. No other nitrate mineral was identified in this research. Calcium nitrate ($\text{Ca}(\text{NO}_3)_2$) is conspicuously absent, despite the availability of calcium in a carbonate setting. This may be due to the greater sensitivity to relative humidity of ($\text{Ca}(\text{NO}_3)_2$) (Hill, 1981).

SEM analysis

SEM-analyses of sediments in the cemented level were uninformative. No distinct mineralogical structures were noted.

SEDIMENTS BELOW THE CEMENTED LEVEL (EXCLUDING DISCRETE, WHITE SEDIMENT POCKETS)

Light Microscopy

Analyses of the sediments below the cemented level (70–75 cmbd and 100–105 cmbd) showed many similarities to sediments above the cemented level. Both samples contain angular calcite grains with micro-fractures, and some grains are coated with clay-sized particles. Angular calcite grains with micro-fractures also are present as isolated particles. Grains composed of a mixture of material also contain smaller particles of calcite. Gypsum occurs as

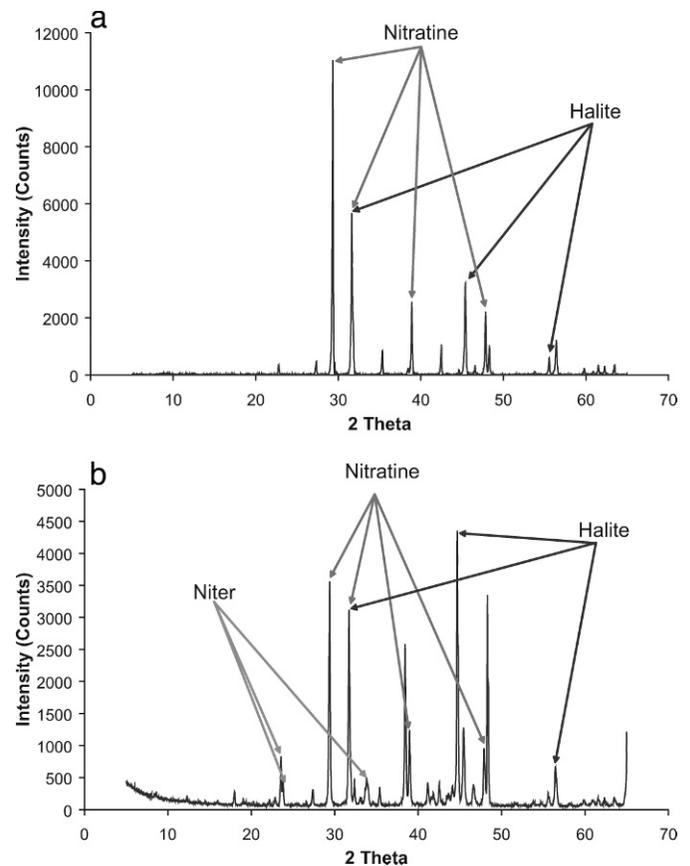


Figure 3. X-ray diffraction (XRD) data. Patterns were generated using Cu K- α radiation. (a) Example of XRD pattern from the cemented level showing the presence of nitratine (peaks marked) (b) XRD pattern of nitratine from the University of Texas collection (peaks marked).

colorless, fine-grained material between larger particles. Bone is also present in these samples.

XRD analysis

The presence of calcite was demonstrated using XRD-analyses in all five samples taken from below the cemented level. Two of the samples show peaks consistent with quartz (70–75 cmbd and 100–105 cmbd), two samples show peaks consistent with dolomite (60–65 cmbd and 87–102 cmbd), and one sample showed peaks consistent with aragonite (87–102 cmbd). Again, microcrystalline particles were present throughout the thin section and quartz may be a constituent of that material.

SEM Analysis

SEM-analyses of sediments below the cemented level were uninformative. No distinct mineralogical structures were noted.

WHITE SEDIMENT POCKETS BELOW CEMENTED LEVEL

Light microscopy and XRD-analysis of the powdery, white sediment showed it to be predominantly composed of

gypsum with some calcite. Energy Dispersion Spectrometry (EDS) analyses on the SEM confirmed the presence of calcium and sulfur. The grains of gypsum did not show any preferred orientation, and grain size did not vary within individual samples or between samples. Smaller detrital materials were scattered throughout the sample, including small fragments of calcite, bone and particles consisting of a mixture of material.

NITRATINE IN CATHEDRAL CAVE

The presence of nitratine in Cathedral Cave provides an additional record of that rare mineral for the state of Nevada. Only four other occurrences of nitratine in Nevada are reported (Castor and Ferdock, 2004). Those records come from Churchill, Elko, Pershing, and Washoe Counties, making ours the first report of nitratine from White Pine County. With the possible exception of the Pershing County record, all of the earlier occurrences were associated with niter, and none have been reported since 1941 (Gianella 1941; not seen, cited in Castor and Ferdock, 2004).

In most cases, the source for the nitrate ion is biological (Hill and Forti, 1997). Nitratine is known to occur as a derivation of bat and woodrat urine (Hill and Forti, 1997), and fossils of these animals occur throughout the excavated section at Cathedral Cave. Those organisms likely played a crucial role with regard to chemical inputs into the cave. If the origin of the nitrate ion at Cathedral Cave is biological, then it is plausible to speculate that other nitrate minerals should be present in the sediments as well. However, that is not the case, and the absence of other nitrate minerals (e.g., $\text{Ca}(\text{NO}_3)_2$) would seem to indicate either a sampling bias and/or a unique enrichment of sodium in the cemented level. Because the XRD data supports the identification of nitratine (NaNO_3), we prefer the latter explanation.

Other potential sources of sodium at Cathedral Cave include other biological origins (non-urine) and dissolution processes. Woodrats are avid collectors of vegetation detritus within a local setting (Betancourt et al., 1990). Saltbush (*Atriplex* sp.) occurs in the vicinity of Cathedral Cave today and did so in the past as well (Thompson, 1979). Species of *Atriplex* are halophilic and will concentrate salts in their leaves in response to salt-stress (Bajji et al., 1998). Detrital material from saltbush enriched in sodium and brought into the cave by woodrats constitutes a potential source of sodium into the cave. Presently, woodrat middens from Cathedral Cave have not been analyzed, but saltbush is known from woodrat middens collected at other localities in Smith Creek Canyon and the Great Basin (Thompson, 1979).

Another possible source of sodium is related to dissolution processes within local bedrock. The source of sodium may be derived from the dissolution of a halide bed. We found no literature references to sulfate or halide beds in the Pole Canyon Limestone, Lincoln Peak

Formation, or Johns Wash Limestone. However, sodium that is not bonded with chlorine can exist in significant quantities in marine carbonates (Land and Hoops, 1973).

It is impossible to state accurately the extent of nitratine in Cathedral Cave. The cemented level constituted a 15 cm interval in a 1 m × 1 m grid, so each of the samples can fall anywhere within a 150,000 cm³ spacing. The occurrence of nitratine was confirmed in two grids. Nevertheless, in all analyses of the cemented level (excluding analyses from the top of the cemented level), nitratine was the dominant constituent. Given the sensitivity of nitratine with respect to relative humidity, its occurrence at Cathedral Cave is potentially informative with regards to paleoenvironmental conditions within the cave.

Nitratine is hygroscopic and deliquescent, and is only stable within a particular range of humidity (Hill, 1981). According to Hill, if relative humidity and temperature exceed a critical value, nitratine “will dissolve and seep into the surrounding sediment or bedrock” (Hill, 1981, p. 131). The instability of nitratine in humid environments calls the timing of the deposition of the nitratine in the cemented level in Cathedral Cave into question.

Several lines of evidence suggest that the nitratine was deposited subsequent to the deposition of other sediments in this horizon. This interpretation is supported by the relatively shallow depth of the nitratine occurrence, and the occurrence of the gypsum pockets that are commonly associated with guano soils (Hill and Forti, 1997). Under this scenario, a likely source for the nitrate and sulfate ions would be bat guano. Similar to deposits of desert caliche, nitrates and sulfates could have been leached from a guano deposit on the cave floor and carried down to some depth where they then re-precipitated at the level encountered during the excavation. The necessary chemical constituents could have entered the system at any post-depositional point relative to non-cementing sediments in this horizon. Although we found no reports of caliche-like layers in cave soils, caliche-like nitrate soils are widely reported from the Atacama Desert in Chile (e.g., Böhlke et al., 1997). This interpretation of the cemented layer would constitute a unique contribution to the science of cave mineralogy.

However, an alternative interpretation is that the deposition and crystallization of nitratine was synchronous with the surrounding sediments and fossils at the level that the nitratine was encountered during the excavation. This suggests a period of low relative humidity that was less than the critical value where nitratine becomes unstable. We assume that low relative humidity in the cave would be associated with arid conditions surrounding Cathedral Cave. Given that the sediments above and below the cemented level lack nitratine, applying a paleoenvironmental explanation for the occurrence or lack of nitratine is enticing. This is particularly so, because one of the primary goals of the paleontological project at Cathedral Cave relates to understanding mammalian response to environmental change prior to the terminal Pleistocene.

Without additional sedimentological or independent support for either hypothesis, it is not possible to resolve the exact timing of nitratine deposition. Our inability to resolve the timing of nitratine deposition highlights the need for future researchers to consider additional excavation and sampling procedures.

SUGGESTIONS FOR FUTURE STUDIES

Because this project was conceived post-excavation, there are several issues and challenges that may improve future studies. The sample sizes and choices were limited to sediments that had not been subjected to a post-excavation, wet-screening process. The on-site, dry-screening of unconsolidated sediment samples prevents any type of quantitative analysis of mineral content and likely contributed to false textures (e.g., clay-sized coatings on grains). Another issue that arose as this project developed was the inability to reconstruct detailed, three-dimensional relationships between samples. Many of the samples for this research were selected from 5 cm levels referenced to 1 m × 1 m excavation square meaning that each sample selected can fall anywhere within a 50,000 cm³ spacing (5 cm × 100 cm × 100 cm). In the context of paleontological studies, excavating in 5 cm intervals is a rigorous protocol. However, for studies that seek to reconstruct paleoenvironmental conditions based on sediment stratigraphy, a much smaller interval would be more informative.

Incorporation of more detailed observations regarding the depositional context (e.g., mineralogy of sediments) of fossils in caves may provide independent and novel data regarding a variety of paleontologically-related questions (e.g., paleoenvironmental, paleohydrology). Near-constant temperatures, varying levels of relative humidity, and isolation from surface weathering are all factors that combine to make cave mineralogy and stratigraphy distinct. In future studies, detailed descriptions of the distribution of soluble elements in the section could help provide insight into the amount of water that has passed through the system.

In the case of Cathedral Cave, the synchronicity of nitratine deposition with vertebrate fossils may have important implications for how Pleistocene mammals interacted with their environment. Independent of the paleontological interpretations, improved understanding of the stratigraphy of cave floor sediments potentially allows for improved interpretations of regional paleoclimatology and paleohydrology.

For future paleontological projects that want to incorporate detailed aspects of sediment stratigraphy, we propose taking small core samples prior to (or in conjunction with) the excavation proceedings. Oriented core samples could be useful for generating cross-sections and chemical/mineralogical distribution graphs. Bedrock samples should also be collected for mineralogical comparisons with excavated sediment sequences. Additionally,

temperature and relative humidity measurements of present-day cave conditions would provide a framework for identifying potential element sources in the context of mineral stability regimes. Explaining the occurrence of minerals (or lack thereof) throughout an excavated sequence may help address a variety of research questions and potentially lead to new ones.

CONCLUSIONS

Multiple methods of analysis indicate some interesting mineralogical occurrences and anomalies in an excavated, paleontological sequence from Cathedral Cave. The presence of nitratine as a cement throughout a discretely excavated level represents only the fifth known record of this arid-climate indicator from the state of Nevada. While the cemented level may preserve a caliche-like deposit leached from guano, the presence of nitratine highlights the potential of detailed, mineralogical studies for providing independent environmental data for paleontological research. Although this project did not follow an ideal methodological path to completion, it serves to illustrate the potential gain from such projects.

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APPENDIX 1.

Summary of sediment samples utilized in this study by material, level, and grid. 'Bulk sediment' represents a sample of loose, unconsolidated matrix irrespective of color. 'White sediment' represents discrete samples of white colored sediments found in isolated pockets below 53 cmbd. 'Bone' samples represent isolated elements that were encrusted with secondary mineral deposition. The type of collection method listed separates those samples collected in the field versus those collected in the lab from dry-screened sediments. TS = Thin Section, XRD = X-ray diffraction analysis, SEM = Scanning Electron Microscopy. If the sample number for a given level exceeds one, the actual number of samples is indicated in parentheses.

Material	Sample Level	Sample Grid	Type	Analysis(es)
Bulk Sediment	25–30 cmbd	1N3E	lab	TS, XRD, SEM
Bulk Sediment	35–40 cmbd	2N3E	lab	TS, XRD, SEM
Bulk Sediment	35–40 cmbd	2N3E	lab	XRD
Bulk Sediment	40–45 cmbd (cemented)	1N3E	field	TS (2), XRD, SEM
Bulk Sediment	40–45 cmbd (not cemented)	2N3E	lab	XRD (2)
Bulk Sediment	45–60 cmbd (not cemented)	2N 2-3E	field	XRD
Bulk Sediment	Cemented	1N3E	field	TS, XRD (2)
Bulk Sediment	Cemented	2N3E	field	TS, XRD, SEM
Bulk Sediment	60–65 cmbd	1N 2E	lab	XRD
Bulk Sediment	60–65 cmbd	1N 3E	lab	XRD
Bulk Sediment	70–75 cmbd	2N3E	lab	TS, XRD, SEM
Bulk Sediment	100–105 cmbd	2N3E	lab	TS, XRD, SEM
Bulk Sediment	87–102 cmbd	2-3E 2N-1N	lab	XRD
White Sediment	65–70 cmbd	1N3E	lab	TS (2)
White Sediment	65–70 cmbd	2N3E	field	TS (2), XRD, SEM
Bone	Varies	Varies	field	TS (2), XRD (2), SEM