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# Analysis of Marine Sediment by Chemical Signatures and Loss-on Ignition to Discover Evidence of Ancient Maya Activities at Site 74, Paynes Creek Salt Works, Belize

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**ANALYSIS OF MARINE SEDIMENT BY CHEMICAL SIGNATURES AND LOSS-ON-IGNITION TO DISCOVER EVIDENCE OF ANCIENT MAYA ACTIVITIES AT SITE 74, PAYNES CREEK SALT WORKS, BELIZE**

A Thesis

Submitted to the Graduate Faculty of the  
Louisiana State University and  
Agricultural and Mechanical College  
in partial fulfillment of the  
requirements for the degree of  
Master of Anthropology

in

The Department of Geography and Anthropology

by  
Kobi Weaver  
B.A., New Mexico State University 2014  
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## **Abstract**

In this thesis, archaeological sediment chemistry, loss-on ignition and microscopic analysis of marine sediment are used to study Site 74 of the Paynes Creek Salt Works in southern Belize. Site 74 was once an ancient Maya salt work. Due to sea-level rise, sea water and mangrove peat now cover the site. Sediment from the site was exported under permit to the Louisiana State University Laboratory. I prepared and delivered the samples to the Louisiana State University Agricultural Chemistry Laboratory for inductively coupled plasma- atomic emission spectroscopy testing (ICP-AES). ICP-AES measured the amount of 20 elements in the sediment. Maps showing variation in the elemental concentrations across the site were made. The study of rapidly-abandoned sites as well as ethnohistorical and ethnoarchaeological theory and methodologies were used to interpret the results. I also performed loss-on ignition to analyze the percent of organic matter in the samples and microscope analysis to analyze the organic composition of the sediment. The sediment chemistry identified areas with long term burning, believed to be from salt-making fires, as well as storage areas. Loss-on ignition was used to identify artifact distribution and spatial use post abandonment, and to show that the yard was most likely not used for salt making. Microscopic analysis positively identified the sediment as red mangrove peat as well as microscopic pieces of charcoal within the sediment in the areas believed to be associated with fires. These tests helped interpret how Site 74 was used, what activities took place there, how the buildings were constructed, the spatial layout of Site 74 and adds to the growing knowledge of ancient Maya salt production at Paynes Creek Salt Works.



## **Introduction**

In this thesis I investigate Site 74 at the Paynes Creek Salt Works, an ancient Maya salt work located off the coast of southern Belize. I evaluate the activities that took place at this location, the spatial layout and usage of the site, and how sea-level rise may have affected the site. The sediment from Site 74 was analyzed using three methodologies. Loss-on ignition was used to determine the percent organic matter of the sediment. Microscopic analysis determined the organic component of the sediment and how that make up differed across the site. Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) was used to test the sediment for 20 elements spatially across the site. This research builds upon previous ICP sediment chemistry analysis at another Paynes Creek salt work (Sills et al 2016). The sediment chemistry is interpreted using ethnoarchaeological and ethnohistorical analogies. Specific attention is given to the study of the modern Maya salt production site of Sacapulas, Guatemala.

Site 74 has pole and thatch buildings which are rarely preserved in the archaeological record. Because of the environmental conditions at Paynes Creek, archaeologists have the unique opportunity to research artifacts that normally degrade. This thesis adds to the knowledge of how the ancient Maya produced salt by using sediment chemistry to describe how the structures were designed, what activities took place in each structure, and how the ancient Maya interacted with Site 74. By studying structures that are rarely preserved in other environments, research at Paynes Creek adds crucial knowledge about the everyday life of the common Maya people that are not often identified in the archaeological record.

## Site 74

Site 74 is one of the Paynes Creek Salt Works in southern Belize and is located in the middle of a coastal salt water lagoon (Figure 1). Based on type-variety analysis performed on pottery and two radiocarbon dates that place the site between AD 660 and AD 770, Site 74 is known to have been occupied in the Late Classic and Terminal Classic periods (AD 600 – 900; McKillop and Sills 2016). The Paynes Creek Salt Works were once an active salt production area in the Maya lowlands where the ancient Maya evaporated brine in pots over fires to produce salt for use and trade with neighboring areas. Due to rising waters caused by climate change, the salt works are now underwater (McKillop et al. 2010; McKillop and Sills 2016). These aquatic conditions provide an anaerobic environment free of oxygen that normally decomposes wood, making Paynes Creek Salt Works one of the few areas where Maya wood is still preserved

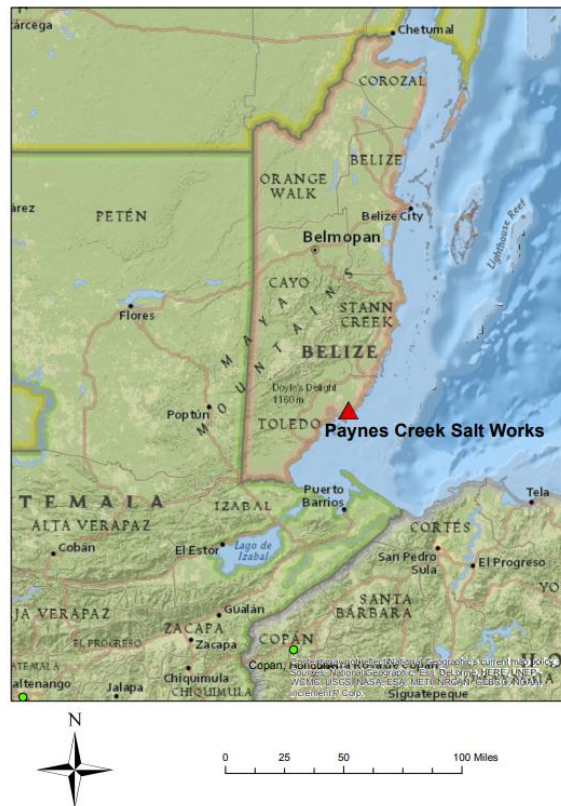


Figure 1. Map showing location of the Paynes Creek Salt Works in Belize.

(McKillop et al. 2010). Such preservation is important in the Maya region because a disproportionate amount of archaeological research has focused on permanent stone structures, and the noble Maya who lived and worked in them.

Site 74 was excavated in 2012 and 2013 by the Underwater Maya Research Group directed by Dr. Heather McKillop as part of an ongoing investigation of the Paynes Creek Salt Works. The site was originally surveyed by identifying and flagging every submerged post. This initial survey exposed two structures and a yard area. Excavations determined that Site 74 consists of two primary buildings, A and B, and a yard. Both structures are made of hardwood posts. The yard is lined with palmetto posts, believed to be a retaining wall (Figure 2; McKillop and Sills 2016). The archaeological team planned and excavated five transects over two field seasons. Transect 1 measured 14 meters in length and was placed along the inside wall of building A. Transects 2, 3, and 4 were placed perpendicular to Transect 1. Transects 2 and 4 extend three meters past Transect 1 along the north and south walls through the center of the

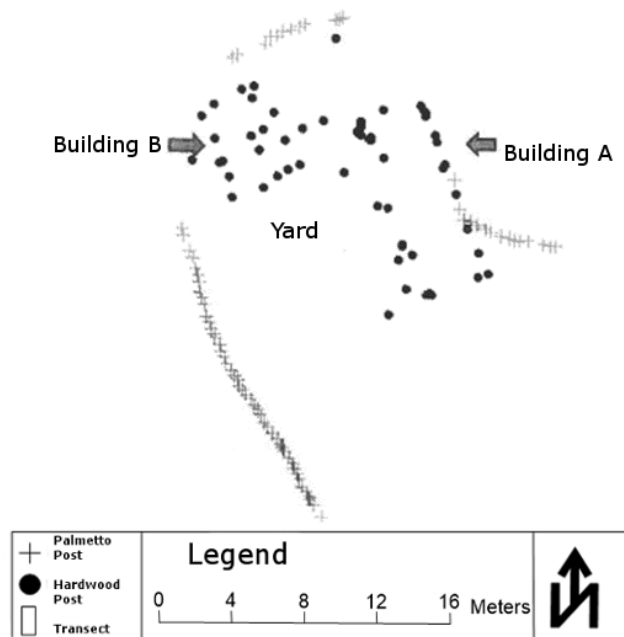


Figure 2. Map showing post location and layout of Site 74. (McKillop and Sills 2016: Figure 2).

room. Transect 3 measures fifteen meters and was placed in the center of the room, out across the yard and one meter beyond the line of palmetto posts (Figure 3; McKillop and Sills 2016).

In building B, Transect 5 was placed along the full length of the room interior of the west wall, extending outside of the room two meters to the north and 1.5 meters to the south. The

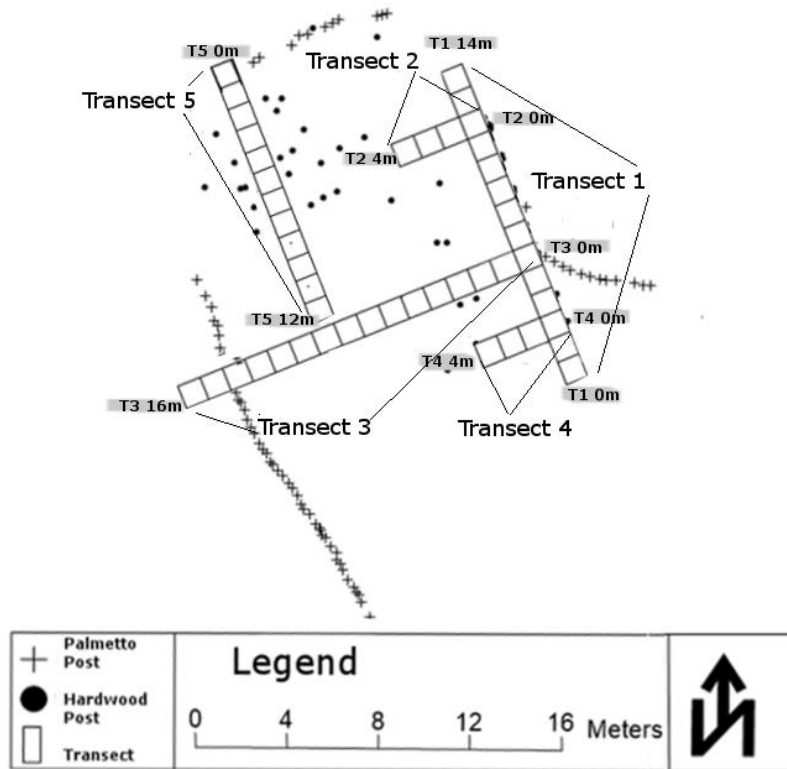


Figure 3. Map showing transects at Site 74. (McKillop and Sills 2016: Figure 3).

transects can be seen in Figure 3 (McKillop and Sills 2016: Figure 3). The transects were placed in such a way that they cover areas from the interior and exterior walls of building A and B, the center of each room, the yard, and extend past the retaining wall (Figure 3).

## Marine Sediment Analysis

Mangroves are tropical coastal trees and shrubs that occur along coastlines, intertidal zones, estuaries and islands (Middleton 2001). These wetland species are an important ecological system as they provide a buffer for coastal communities against hurricanes, provide food and habitat for marine life and act as a filter of ocean water and sediment (McKee et al. 2007). There are three kinds of mangrove: red, black and white. Red mangrove, *Rhizophora mangle*, is used by scientists to study sea-level rise because of the unique ability red mangroves have to grow and adapt to sea-level change (McKee 2004; McKee et al. 2007; Middleton 2001; Toscano and Macintyre 2003). Red mangroves have two distinct types of roots: aerial roots that occur above the water and allow oxygen to enter the organism, and below-ground roots that grow in an anaerobic environment (McKee 2004).

As sea levels rise, red mangrove's growth outpaces rising water levels to keep leaves above the water. This growth pattern allows the mangrove to obtain sunlight for photosynthesis. The mangrove roots add organic matter to the sediment to keep the ocean waters from eroding away the existing peat and sediments in which mangroves live (McKee and Faulkner 2000). Peat formation in the mangrove forests of Belize is due to the deposition of mangrove roots in the sediment and not above ground flora like twigs and leaves that entered the system (Middleton and McKee 2001). During times when sea-levels rose, mangrove roots experienced rapid growth to help propel the plant upward. During times when the sea-level fell, the root systems stop producing roots (McKee et al. 2007). Loss-on-ignition will determine how much of a sediment sample is composed of organic material. By determining the amount of fine mangrove roots in a sediment sample, one can determine if the mangrove plant had to deposit many roots in the sediment to contend with sea-level rise. If a sample has a high percentage of fine red mangrove

roots, it originated when sea levels rose and roots were deposited in the soil to help anchor the plant and allow rapid growth to keep the mangrove leaves above the water.

### **Chemical Analysis of Anthropomorphic Sediment**

Anthrosols and anthropomorphic sediment are terms for soil or sediment that have been chemically altered by human activity over time (Wells et al. 2002). Friesem (2016) describes two different processes of site formation: natural processes and cultural processes. Within cultural processes, there is a time line of site formation starting at “before occupation” to “human occupation”, “abandonment”, “post abandonment”, and finally the “archaeological excavation.” From human occupation to post abandonment, human activities chemically alter soils and leave behind signatures that can be tested and analyzed during the excavation phase.

The testing of anthrosols is often referred to as soil chemistry. It was pioneered by Olof Arrhenius for archaeological use in 1926 when he came across data he could not explain while studying how plants absorb nutrients in relation to hydrogen content. There were several areas in the estate he was studying that had high concentrations of phosphate, even though they were in an area with consistently low phosphorus levels. There were no known reasons why, seemingly sporadically, the phosphate levels spiked. Arrhenius (1963) studied this phenomenon for almost forty years before publishing “Investigation of Soils from Old Indian Sites,” in which he was able to correlate the areas of high phosphorus with the dwellings of ancient Viking farms and settlements which had been abandoned and destroyed. Even though there were no artifacts or ancient architecture of the Vikings left to find, physical evidence of their activities remained within the ground on which they lived. In this case, the chemical signatures were used to locate an archaeological site that was no longer visible. With the advancement of soil science, the

interpretation of chemical signatures can be used for locating archaeological sites as well as interpreting activities. There are three main methods for understanding how chemical signatures and ancient activities correlate: ethnoarchaeology, ethnohistory and the study of rapidly abandoned sites (Terry et al. 2004).

### **Ethnoarchaeological and Ethnohistorical Studies**

Ethnography in the anthropological context is the study of people and culture. Ethnography is done by studying one aspect of culture in great detail. Anthropologists use ethnography to record and study living people. However, ethnographic methodologies can be used to study ancient peoples as well. Ethnoarchaeology is the study by archaeologists of peoples and their material culture to inform archaeological research. Ethnoarchaeological studies of soil involve directly observing the activities and spatial use of modern peoples, seeing what chemical signatures are left behind in the soil and then comparing them to the ancient soil signatures. For example, Luis Barba (2007) studied the homes of modern Maya villagers who live a traditional lifestyle in Tlaxcala, Mexico. He focused on the chemical signatures of waste disposal, food storage, consumption and preparation, and paths. Over the course of his study, Barba (2007) identified chemical signatures for sleeping, eating, animal housing and paths. Barba also identified that highly-traveled areas were low in phosphates whereas areas dealing with food preparation and consumption had high phosphate levels. Fernández et al. (2002) also studied the homes of modern Maya who lived in pole and thatch buildings with earth floors. The researchers noted the same chemical patterns as Barba. Both studies found low levels of phosphorus on paths and patios and high phosphorus levels in gardens and food consumption areas. Both also noted wood ash produced a correlation between phosphorus and potassium.

Middleton and Price (1996) used ethnoarchaeological methods when they analyzed the soil chemistry in a modern household and compared that with the soil chemistry in an archaeological household in Oaxaca, Mexico as well as an archaeological house in British Columbia, Canada. The researchers found the same chemical signatures for cooking and occupation in both the modern and archaeological homes. The same chemical signatures were found in both ancient homes in Mexico and Canada. Middleton and Price (1996:681) noted that chemical signatures “form independent of the natural soil formation and soil matrix.”

Outside of the Maya region, ethnoarchaeological methods also are used. Knudson (2004) spent time at multiple arctic fish camps studying the activities which were performed there and how the activities were performed, as well as collecting soil samples to identify chemical signatures of fish processing. Linselotti and Madella (2012) conducted an ethnographic study in northern India on dung usage and the chemical evidence dung leaves behind for archaeological purposes.

Ethnohistorical studies involve reading historical documents to help inform archaeological research. An example of an ethnohistorical study is reading documents written by Spaniards during the contact period to gather information about how Maya homes were built. Although bias and false information may be included in these documents, looking at historic records with a discerning eye can still provide valuable information to archaeologists.

#### The Sacapulas Salt Makers: A Case Study in Maya Salt Production

In relation to Site 74, an ethnographic study of salt production will be used to inform my research about the chemical signatures of salt production. An ethnographic study by Reina and Monaghan (1981) was performed in 1978 on the Sacapulas Maya of Guatemala who had been



making salt in a traditional manner long before Spaniards came to the Americas. Reina and Monaghan's research included studying historical documents and performing a traditional anthropological field study of the salt-making process.

Colonial documents written by Spaniards detail salt production in the region. In 1629 Alfonso Tovilla wrote in detail about the production of salt by the Sacapulas. Although the methods used in Guatemala and the ones used at Paynes Creek vary upon the source of the brine -sea water vs salt flats- there are still similarities that can be seen in the evaporation and forming of the salt. In the first step of salt production, the Sacapulas Maya harvest soils from the salty playas which are high in natural sodium. The harvested soil is spread flat and allowed to dry before fresh water is poured over it. The water pulls salt from the playa into the harvested soil. The salt stays on top of the harvested soil as it dries. This process is repeated for several days before the harvested soil is completely saturated with salt and no more can be lifted from the playa. The salty soil is then gathered and brought to the kitchens for refinement (Reina and Monaghan 1981). Next, the salt saturated soil is placed in a wooden refinement box called a cajon. The cajon is a wooden box with a straw mat filter on the bottom. Water poured into the cajon dissolves the salt, allowing it to pass through the straw filter while leaving the soil behind and filling pots beneath with the newly created brine. The Sacapulas Maya refined the brine into salt by setting clay pots filled with brine over the fire and evaporating the water to form salt cakes (Reina and Monaghan 1981:26).

The kitchens are described as enclosed 6x8 meter structures not used for residential purposes and dug slightly into the ground. There is a roof gap where smoke can escape. They were described by anthropologists as being littered with broken pottery. Along the inner walls the salty soil is piled up and pots are filled with processed salt, firewood and ash. In the center of

the kitchen is a 1x2 meter platform of packed earth used to cook the pots of brine. The brine spills over the pots as it evaporates. The fire is continuously stoked. The white salt water from the soil brine floats to the top and is scooped out and placed into other pots to continue evaporating. The Sacapulas Maya make two different kinds of salt: white and black. Black salt contains the mineral apthitalite,  $(K,Na)_3Na(SO_4)_2$ , which is not found in white salt. The black salt is more highly valued. The anthropologists noted that in two days, a two-person team produced about two hundred and fifty pounds of salt (Reina and Monaghan 1981:29).

This case study is important for informing my work on Site 74 because it gives some insight into how the salt-manufacturing buildings may have been configured and how they may have functioned. Although Paynes Creek and the Sacapulas Maya differed in their methods of brine collection, what we know of salt production is similar to the ethnographical study observed here, most notably in the setup of the fire and pots, or briquetage. The placement of the fire in the center of the building, the common occurrence of brine spilling over, as well as the presence of salt and stacked wood along the inside of the enclosed structures provide, some idea of what chemical signatures may have been left behind in kitchens. The patterns observed at Sacapulas match the chemical analysis performed on the Chan b'i salt works by Sills, Wells and McKillop (2016). Chan b'i is another site within the Payns Creek Salt Works. At Chan b'i high levels of phosphorus, sodium, and potassium accumulated in the middle of the room where the fire would have been (Sills et al. 2016).

### **Rapidly Abandoned Sites**

In addition to ethnoarchaeological and ethnohistorical studies, another way archaeologists have determined the chemical signatures of ancient activities is by looking at ancient areas that

were abandoned so quickly that ample artifact evidence remains to reconstruct activity patterns. These places were left so rapidly that the residents did not pack up, clean or remove artifacts. The site would then need to be preserved in such a way that distinctive activity areas could be observed, then tested for chemical signatures. One of the most famous examples of such a site is Pompeii, where Mount Vesuvius erupted and trapped the people of Pompeii, and their belongings, in layers of ash. A similar site exists in the Maya world: Joya de Cerén in El Salvador. Cerén was an ancient Maya village that was rapidly abandoned because of a volcanic eruption around AD 600. Cerén has been preserved in a state that allows archaeologists to study the artifacts and the “behavioral processes that link them” (Parnell, Terry and Sheets 2002:333). Soil science was conducted at Cerén, specifically looking for phosphorus and heavy metals. When coupled with in situ artifacts, areas used for food preparation, waste, and pigment storage were found and the distinctive chemical signatures marked for future research (Parnell, Terry and Sheets 2002).

Another city in the Maya area that shows rapid abandonment is Aguateca, Guatemala, which was depopulated in the early ninth century AD due to warfare that caused the residents to flee, leaving their homes and belongings behind (Terry et al. 2004). The soil chemistry performed at Aguateca aided in the location of food preparation areas and two distinct types of middens. One type of midden was used for disposing mostly of food waste, as identified by extremely elevated levels of phosphorus and nitrogen. Two others middens were for disposing of waste relating to craft production, as identified by the presence of heavy metals (Terry et al. 2004). Distinct chemical signatures for different types of middens were also found in El Coyote in Honduras. Here, Wells used ICP-AES on the plazas and adjacent disposal areas to identify the chemical signatures for food waste, lithic waste and ritual waste (Wells 2004).

One of the strengths of soil science is that it can be used in conjunction with artifacts to build a deeper understanding of how a space was used. Soil science can also be used in cases where few physical artifacts remain, or where the artifacts that are present do not represent how the space was used due to poor preservation or disturbances (Terry et al. 2004). Most Maya settlements were abandoned gradually. The residents had the time to remove their belongings or move them in such a way that the recovered artifacts do not always accurately represent how the space was used. Using soil analysis on floors allows archaeologists to more accurately reconstruct activity patterns than by relying on artifact patterns alone because the spatial patterns of chemical residues that have been absorbed into the floors closely reflect how the space was used in the past (Fernandez et al. 2002; Parnell, Terry and Sheets 2002).

### **Common Anthropomorphic Chemical Signatures**

Chemical signatures have been identified with specific human behaviors in the Maya area and elsewhere. Phosphorus (P) is the most common element that is indicative of human-environment interaction. Phosphorus can be found as evidence in activities such as food processing, consumption and disposal, burning wood, fish processing, gardening and dung disposal (Fernández et al. 2004; Knudson and Frink 2010; Linselotti and Madella 2012; Middleton and Price 1996; Terry et al. 2004). Due to phosphorus's highly reactive nature, P is rarely found pure in nature (Iheagwara et al. 2013). As organic materials decompose, the phosphorus becomes fixed into phosphate. Although the two terms are often used interchangeably, phosphorus is a pure element whereas phosphates are ions made of a single phosphorus atom bonded with four oxygen atoms. These ions often bond with other elements. In soil, phosphates commonly bond with calcium, iron and aluminum (Iheagwara et al. 2013;

Parnell et al. 2002). When testing for phosphorus, phosphates are included; thus both terms are used (Iheagwara et al. 2013).

In enclosed spaces, phosphorus levels are lower inside the room and higher around the edges if waste is swept to the sides or piled up during cleaning and sweeping (Middleton and Price 1996). Many human activities do not leave any traces of phosphorus behind. Phosphorus can also occur naturally in the environment. Due to the natural abundance of phosphorus and growing scientific advancements used to accurately measure the levels of different elements such as ICP analysis, archaeologists now use a multi-element analysis of anthrosols (Holliday et al. 2010; Wilson et al. 2008).

Potassium (K) and magnesium (Mg) are also found in conjunction with phosphorus at food processing and consumption sites (Fernández et al. 2004; Middleton and Price 1996). Heavy metals such as copper (Cu), iron (Fe), mercury (Hg), manganese (Mn), lead (Pb), and zinc (Zn) are indicative of pigment and craft production (Parnell et al. 2004). Strontium (Sr) barium (Ba) and calcium (Ca) in raised levels can result from deposits of marine shells (Sills et al. 2016).

Middens often have higher phosphorus levels because of the quantities of decomposing organic matter. Different middens produce distinctly different chemical signatures. For example, stone tool production middens should have high concentrations of iron, titanium, aluminum and potassium (Wells 2004). The most common chemical signatures can be seen in Table 1.

Table 1. Anthropomorphic Activities and Correlating Chemical Signatures		
Human Activity	Elevated Chemical Concentrations	Reference
Food Processing and Consumption	Phosphorus, Potassium, Magnesium	(Fernández et al. 2004; Middleton and Price 1996)
Food Disposal	Phosphorus	(Terry et al. 2004)
Pigment and Craft Production	Copper, Iron, Mercury, Manganese, Lead, Zinc	(Terry et al. 2004)
Enclosed Spaces	(Phosphorus is removed due to sweeping)	(Middleton and Price 1996)
Gardens	Phosphorus, Zinc, low PH	(Fernández et al. 2004)
Fish Processing	Sodium, Phosphorus, Potassium, Magnesium	(Knudson and Frink 2010)
Dung	Aluminum, Barium, Calcium, Chromium, Copper, Iron, Magnesium, Molybdenum, Nitrogen, Lead, Phosphorus	(Lanselotti and Madella 2012)
Wood Ash	Potassium, Magnesium, Phosphorus	(Fernández et al. 2004; Middleton and Price 1996)
Marine Shell Deposits	Strontium, Barium, Calcium	(Sills et al. 2016)
Lithic Middens	Iron, Aluminum and Potassium	(Wells 2004)
Salt Production	Phosphorus, Magnesium, Potassium (In correlation with briquetage)	(Sills et al. 2016)

## **Inductively Coupled Plasma Analysis and Underwater Sites**

Chemical testing has been used all round the world on different types of soils, including sediment from underwater sites. A markedly less common use is chemical analysis on sediments for archaeological purposes. Most recently for the Maya region Sills et al. (2016) were the first to use Inductively Coupled Plasma Mass Spectrometry ICP-MS to analyze the sediment of an underwater site at another site in the Paynes Creek Salt Works in southern Belize.

ICP, or Inductively Coupled Plasma, is an analytical technique that is used for quantifying elemental constituents within a substance. There are three different kinds of ICP testing: Inductively Coupled Plasma- Optical Emission Spectrometry (ICP-OES), Inductively Coupled Plasma- Atomic Emission Spectrophotometry (ICP-AES) and Inductively Coupled Plasma- Mass Spectrometry (ICP-MS). All techniques use the ICP to super heat the sample until the atoms become ions, then the OES, AES or MS sort and detect the different ions (Tyler 2011). ICP techniques all have high detection limits that can determine into the parts per trillion (some in parts per quadrillion) range for samples. They are therefore ideal for analyzing samples in which detecting ranges in elements is important (Tyler 2011). For example, the sites at Paynes Creek Salt Works are all inundated with sea water. The sea water has high levels of sodium. All samples taken from this site will also have high sodium levels because of the sodium from the sea water. In order to determine meaningful variation in sodium levels to study the anthropomorphic changes to the sediment, the method for chemical analysis needs to be sensitive to a range of variation.

The need for accurate variation detection led to the use of ICP-MS when Sills et al. (2016) analyzed the anthropomorphic sediment at the site of Chan b'i. This thesis builds upon that research by analyzing the sediment at Site 74, another salt production site within the Paynes

Creek Salt Works. I evaluate and map the chemical patterns revealed by ICP-AES at Site 74. I then compare the chemical patterns with published artifact analysis and spatial analysis of the buildings at Site 74 (McKillop and Sills 2016). The chemical analysis from Site 74 is then compared to the chemical signatures and patterns found at Chan b'i to learn more about Site 74 and the Paynes Creek Salt Works.



## Research Questions

This research takes a three-step approach to analyzing the sediment Site 74. Loss-on ignition was performed to determine the percent organic matter (%OM) of the sediment and to explore how sea level may have affected the site. The sediment was examined under a microscope to analyze the organic composition of the sediment and how the composition varied across the site. Lastly, I submitted sediment samples to the Louisiana State University Agricultural Chemistry Laboratory for inductively coupled plasma analysis (ICP-AES). I evaluate the results of twenty elements to interpret what activities may have taken place where at Site 74. These tests will help me analyze Site 74 holistically to determine how the site was used spatially, for what purposes, and how natural and human processes affected the site.

I expected the percent organic matter to be high throughout the site, with the highest being outside the site parameter. I hypothesized that this pattern was due to the line of palmetto posts acting as a retaining wall and that mangrove peat would have begun to form a layer earlier outside of the wall. I expected the microscopic sorting to have high amounts of both coarse and fine mangrove roots as well as high amounts of charcoal throughout the site. The charcoal would come from the fires used to evaporate brine to produce salt. In conjunction with areas of high charcoal, I hypothesized that the sediment chemistry would show chemical evidence of consistent burning. I also believed that evidence of walls along the structure would be present from a buildup of chemicals due to sweeping debris away from the center of the room. I also expected the sediment chemistry to show evidence of salt production and storage with large variation in levels of sodium, potassium and phosphorus.

## **Materials and Methods**

### **Excavations**

Excavations were carried out at Site 74 in 2012 and 2013 by the Underwater Maya Research Program. Transects were placed along the interior wall of each building and extended two meters beyond building A and B in each direction. Two other transects were placed in building A along the north and south walls. A transect was also placed to cut across the yard and extend one meter outside the site. A layer of mangrove peat was removed so excavations could start on the floor the Maya would have stood upon. The transects were excavated in 10 cm intervals to a maximum depth of 60 cm. Stainless steel blades were used to cut the mangrove peat into sections and then the sections were removed. For the column sample, each section was cut, removed and wrapped in cling wrap. The cling wrap was labeled to indicate which side was the top (McKillop and Sills 2016).

### **Loss-On Ignition**

Loss-on ignition is used to measure the amount of organic matter in soils and sediments. The percent of organic material in the Site 74 sediment was determined using the same loss-on ignition procedures used in other studies at the Paynes Creek Salt Works (McKillop et al. 2010). The sediment samples were excavated by Heather McKillop and the Underwater Maya Research Group, sealed in whirlpack bags, labeled, and exported under permit to the Archaeology Lab at Louisiana State University. There, the samples were stored in a refrigerator to prevent them from molding. The procedure for loss-on ignition is as follows.

Approximately five grams of each sample were taken from the bag using a clean plastic spoon. The samples were placed in an aluminum tin. The tin was labeled with the site number, transect number and meter number on the side and bottom. Then the tins were placed in the drying oven at 60 °C to dry overnight (Figures 4-6).



Figure 4. Photo of samples in drying oven.



Figure 5. Photo of wet sample.



Figure 6. Photo of dry sample.

The crucibles, lids, mortar and pestles were sterilized by washing in a 1% hydrochloric acid (HCL) solution and then air dried or dried with kimwipes (Figure 7). The crucibles were labeled 1-6 with blue acrylic paint (Figure 8). Each of the labeled and empty crucibles were weighed. The weights were recorded. After drying overnight, the tins were removed from the oven. Each sample was then placed in a sterile mortar and ground using a pestle. Pieces of rock and shell were removed using sterilized tweezers and sieves. The ground sediment was placed in the crucibles and weighed. The crucibles were placed in the desiccator filled with dryrite



Figure 7. Photo of washing crucibles with HCL solution. (Photo by Cheryl Foster)

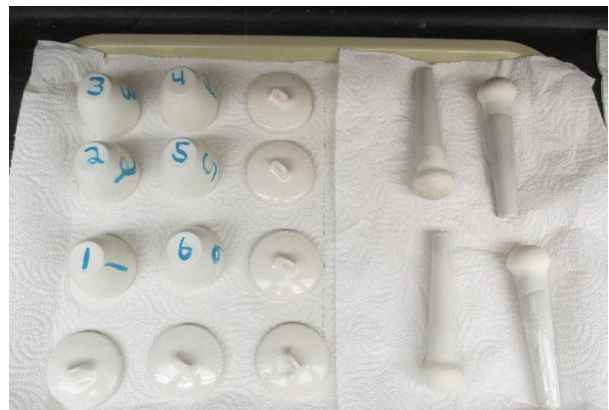


Figure 8. Photo of labeled crucibles.

overnight (Figure 9). The dryrite prevented the samples from absorbing moisture and removed excess moisture from the samples. The next day, the crucibles were placed in the muffle furnace at 105°C for four hours (Figures 10-11). After four hours, the crucibles were removed and the weights recorded. Crucibles were placed back in the oven at 600°C with the lids. Samples with higher amounts of organic matter take longer to fully burn and thus the crucibles were in the oven until the sample was fully ashed, between six to ten hours (Figures 12-13). The change in color and texture the sample undergoes during this process can be seen in Figure 14.



Figure 9. Photo of desiccator.



Figure 10. Photo showing furnace at 105 °C.



Figure 11. Photo of the inside of the furnace at 105 °C.

After the samples had finished the second burn, they were removed from the furnace and placed in the desiccator to cool. The crucibles were weighed. The change of weight over the entire loss-on ignition can be seen in Figure 15. The data were entered into an Excel spreadsheet and the formula developed by an Underwater Maya Research Group collaborator Karen McKee (2004) was used to determine percent organic matter and can be seen in Table 2.



Figure 12. Photo showing furnace at 600 °C.



Figure 13. Photo of the inside of the furnace at 600 °C.



Figure 14. Photo showing the weight of sample 2 throughout the loss-on ignition process.

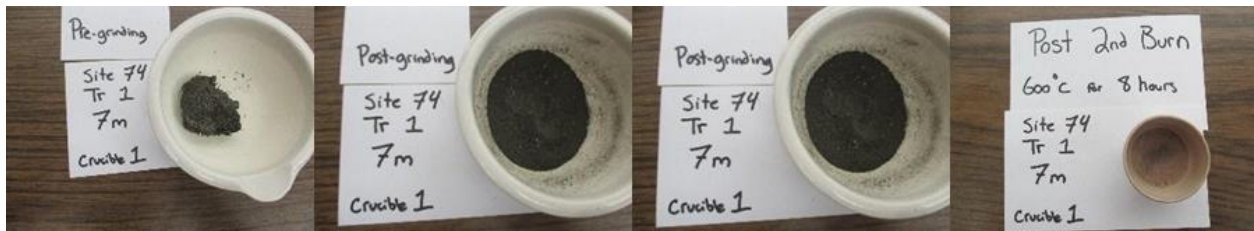


Figure 15. Photo showing texture and color of sample 1 throughout loss-on ignition

Table 2. Formula for Determining Percent Organic Matter (%OM)
$\%OM = [(W_{105} - W_{400})/W_{105}] \times 100$ <p>where: <math>W_{105}</math> = weight of soil at 105° C</p> <p><math>W_{400}</math> = weight of soil at 400°</p>

### Microscopic Sorting

Microscopic sorting was used to visually identify if the sediment sample was red mangrove peat using methods developed by Underwater Maya Research Group member and mangrove scientists Karen McKee (Figure 16). An equal amount of each sample was sorted, a flat one eighth of a teaspoon, and was taken from the whirlpack bag using a clean measuring spoon. The sample was then placed in a 250µm geological sieve and thoroughly rinsed. The sample was then placed in a petri dish filled with water. Under the microscope the sample was classified and sorted. The sample was sorted into five vials using tweezers: coarse red mangrove roots, fine red mangrove roots, organic, inorganic, and unknown (Figure 17). The fine red mangrove roots vials contained fine red mangrove roots. The coarse red mangrove roots vial contained coarse red mangrove roots. The organic vial contained charcoal, leaves and wood. The inorganic vial contained shell and quartz. The unknown contained unidentified items. Fine and coarse red mangrove roots were collected separately because fine roots are resistant to decay and can be used for carbon dating (McKee et al. 2007). Figure 18 shows the difference between fine and coarse red mangrove roots as seen under the microscope.



Figure 16. Photo showing the microscopic sorting process (Photo by Cheryl Foster.)



Figure 17. Photo showing the vials used for microscopic sorting.

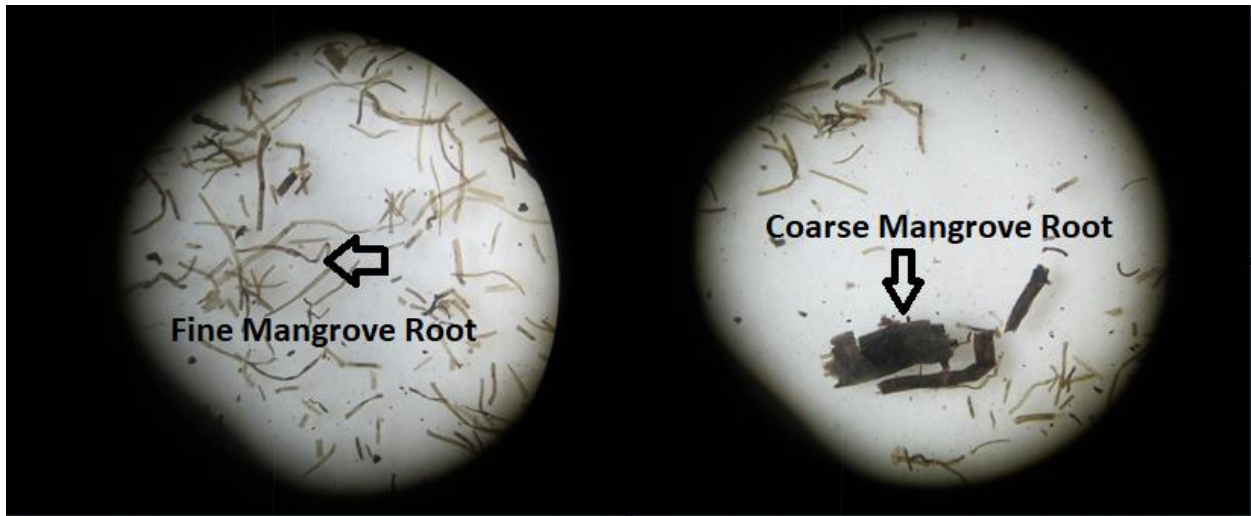


Figure 18. Photo showing the difference between fine red mangrove roots (left) and coarse red mangrove roots (right).

After the sample was sorted, the coarse and fine red mangrove roots were individually weighed. This was done by weighing a 300 $\mu$ m mesh. The mesh was placed over the 250 $\mu$ m geological sieve. The vial containing the roots to be weighed was drained over the mesh. The mesh with the roots was dried on a paper towel and weighed in grams. After the weight of the mesh was subtracted, the weight of the roots remained.

### **Sample Preparation for ICP-AES Analysis**

Fifty-four samples from Site 74 were taken from refrigerated storage in the Archaeology Laboratory at Louisiana State University and sampled. A clean plastic spoon was used to remove approximately fifteen grams of sediment from the larger plastic whirlpack storage bags. The samples were then placed in individual whirlpack bags labeled as followed: Site 74 Transect # Meter # (Figure 19).



Figure 19. Photo showing sample prepared for delivery to the Louisiana State University Agriculture and Chemistry Laboratory.

### **Microwave Digestion**

An extraction process is performed on a sample before ICP-AES can be completed. The extraction process breaks down the elemental bonds in a sample to allow the individual elements to be counted. The Louisiana State University Agricultural Chemistry Laboratory uses Microwave digestion EPA 3052. This method places 0.5g of the sample in 9mL of nitric acid and 3 mL hydrofluoric acid, seals the container and microwaved them for 15 minutes. The temperatures reach about 180 C in less than 5.5 minutes and remain there for the rest of the time. The sample is then cooled and separated by filtration, centrifuged or allowed to settle and separated by hand. It is then ready to be analyzed via ICP-AES.



## **Inductively Coupled Plasma- Atomic Emission Spectroscopy**

The Louisiana State University Agricultural Chemistry Laboratory uses the Environmental Protection Agency's ICP-AES Method 200.7, Revision 4.4 in their lab. This ICP analysis involves multiple pieces of equipment, the most important being the nebulizer, plasma torch and spectrometer. The sample, after having gone through microwave digestion, is placed into the AES machine where a nebulizer turns the sample into an aerosol mist and mixes it with argon gas. This mixture is then pumped into a pressurized chamber where the largest particles fall to the bottom and the smallest particles, about 1% of the total sample, move into the torch. The sample is then injected into a plasma torch where it burns at about 10,000 K. Heating the sample excites the electrons in the sample and the electrons emit energy in the form of light. Each element has a unique wavelength, or turns a specific color, when the electrons are excited. An optical spectrometer then measures and records the wavelengths given off by the burning sample and matches them to the corresponding elements. The element totals were measured in parts per million (PPM) and as a percent. I converted all numbers into PPM for consistency.

## **Mapping Element Concentrations**

After receiving the ICP-AES results from the LSU Agricultural Chemistry Laboratory, I created 20 element distribution maps using Surfer 15 mapping software. Mapping was done by creating "post maps", where a "post" represented the location a sediment sample was taken. The element's value was assigned to each post. A contour was created that showed how the levels of each element changed within the transects.

## Results

### Element Concentration Maps

All the following elemental concentrations are reported in parts per million (PPM). The color variation shows areas of higher and lower concentration. The white or light yellow marks an area with little to no amount of the given element. The color orange shows medium amounts of the given element. Dark red show high levels of the element.

Aluminum levels are elevated along the east wall of building A. Low levels appear in the yard and the rest of the site has medium aluminum levels (Figure 20).

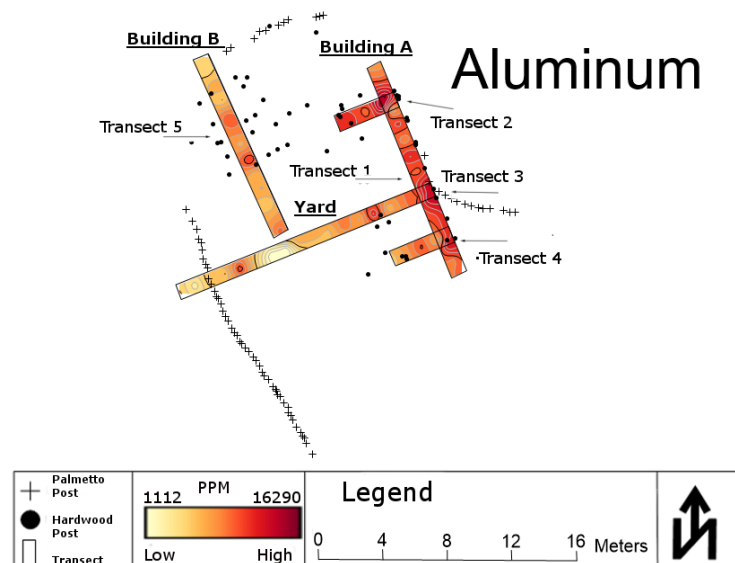


Figure 20. Map of aluminum concentrations.

Arsenic levels are high throughout the entire site, specifically along the North and South walls of building A. The arsenic levels are slightly elevated outside of the site boundary (Figure 21).

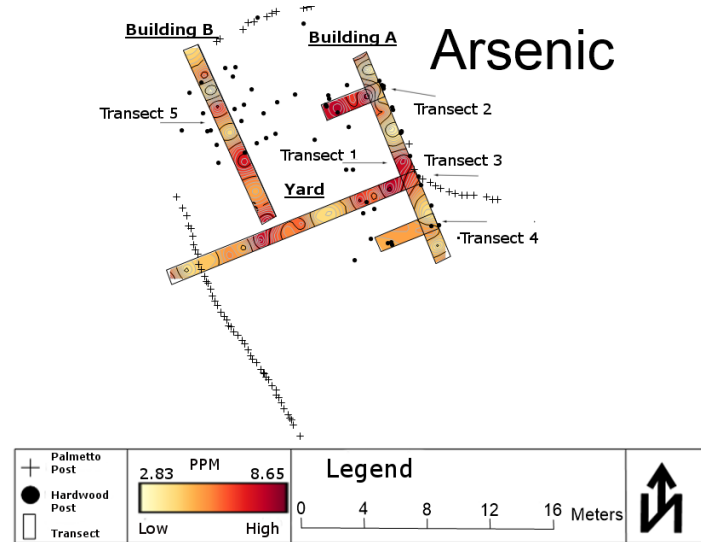


Figure 21. Map of arsenic concentrations.

Barium levels are low in building B and medium in building A with a spike along the north-west wall. Levels in the yard are low, with a few spikes at the east end. Barium levels are low outside the site (Figure 22).

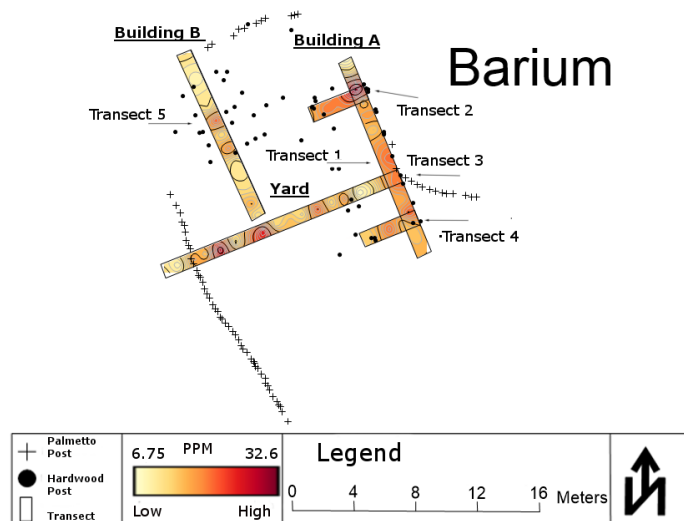


Figure 22. Map of barium concentrations.

Boron has high variation throughout Site 74. The highest levels are in the middle of building B and in the southeast corner of building A. The yard has low levels of boron. The lowest levels are outside site (Figure 23).

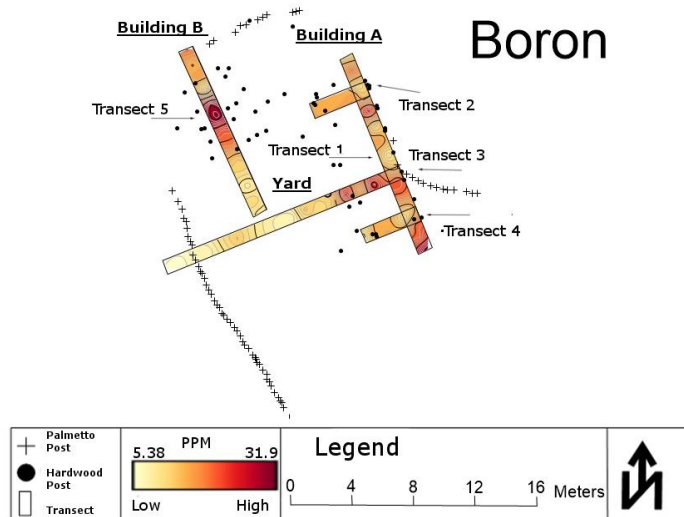


Figure 23. Map of boron concentrations.

Cadmium levels are low throughout the site with the notable exception of the northwest region that encompasses the north wall of building B and part of the yard before the north retaining wall (Figure 24).

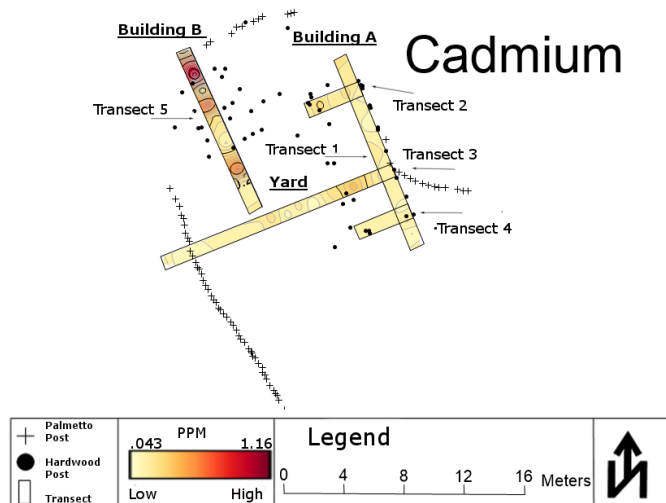


Figure 24. Map of cadmium concentrations.

Calcium levels are medium-low throughout most of the site but spike in the yard at the end of Transect 5, along the north-east wall of building A and medium along the South-East wall of building A (Figure 25).

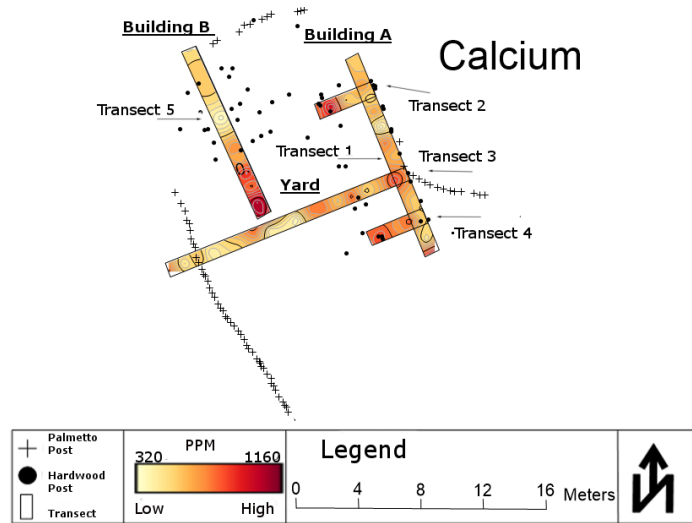


Figure 25. Map of calcium concentrations.

Chromium levels are high throughout the site, specifically along the west wall of building A, and spike in building B and the yard. The levels drop outside the site boundaries of palmetto palm posts (Figure 26).

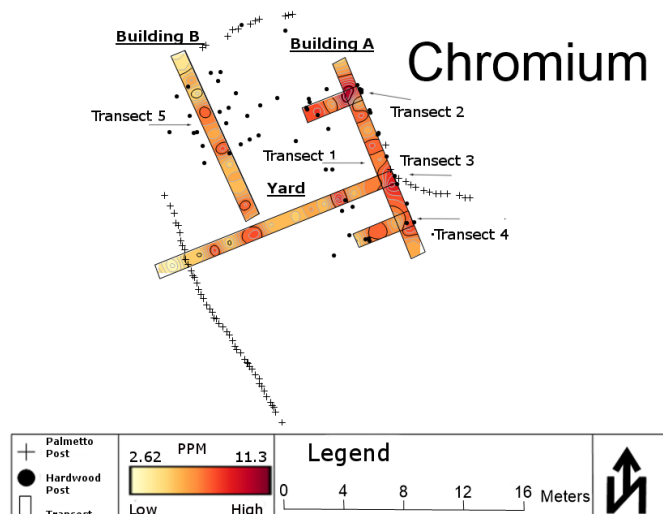


Figure 26. Map of chromium concentrations

Cobalt shows extreme variation throughout Site 74, spiking at the north end of building B, the middle of the yard, and throughout building A. Cobalt levels drop outside of the site boundary (Figure 27).

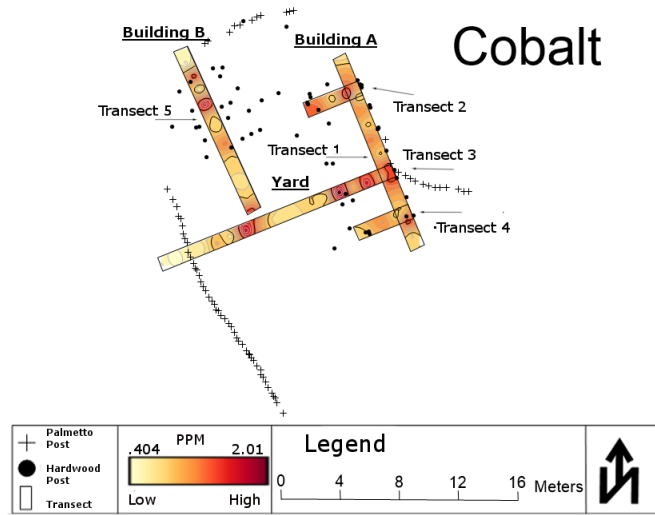


Figure 27. Map of cobalt concentrations.

Copper levels are low throughout the site, apart from the single elevated area in the middle of building A (Figure 28).

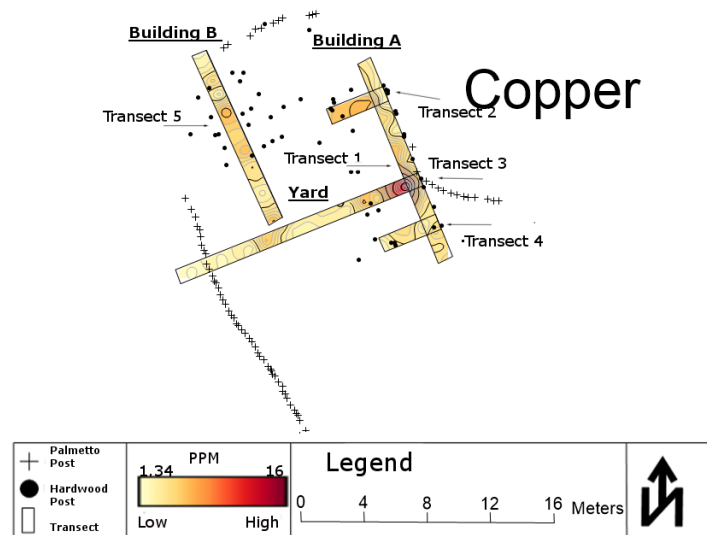


Figure 28. Map of copper concentrations.

Iron levels are high throughout the site, but fall to almost nothing outside of the site boundary marked by palmetto palm posts. The highest lead levels are concentrated in building A (Figure 29).

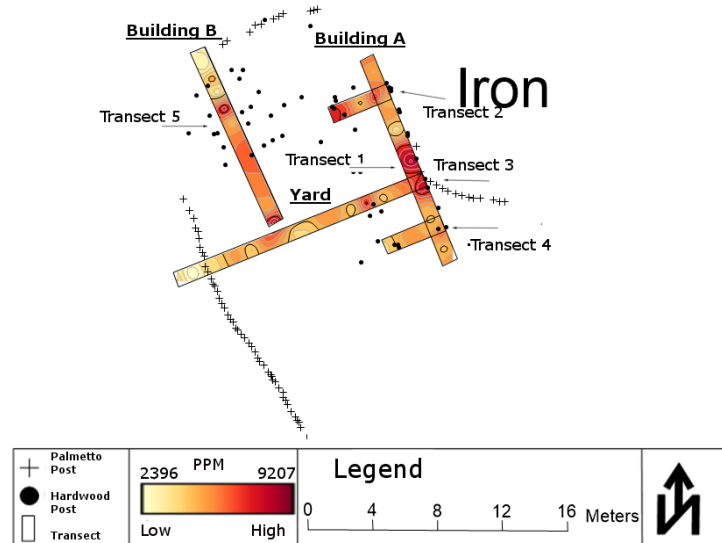


Figure 29. Map of iron concentrations.

Lead levels are medium throughout Site 74, but fall outside of the site boundary. The highest lead levels are concentrated in northeast section of building A (Figure 30).

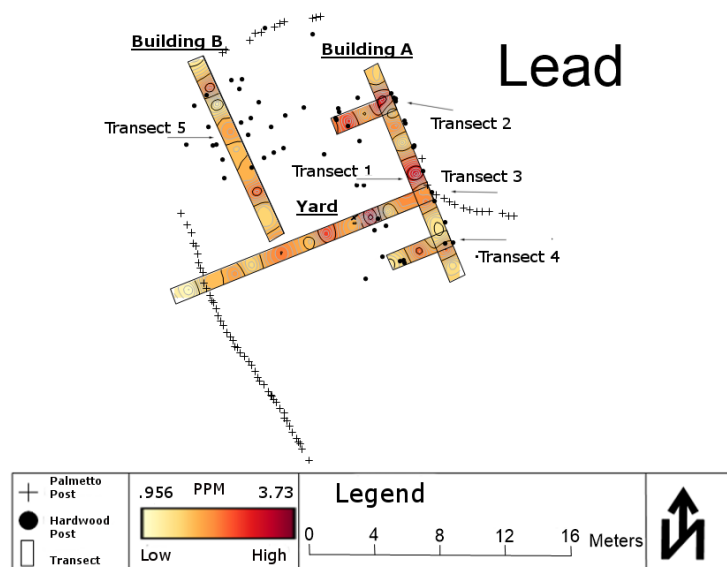


Figure 30. Map of lead concentrations.

Magnesium levels are low throughout the site except for a single spike in the middle of building B (Figure 31).

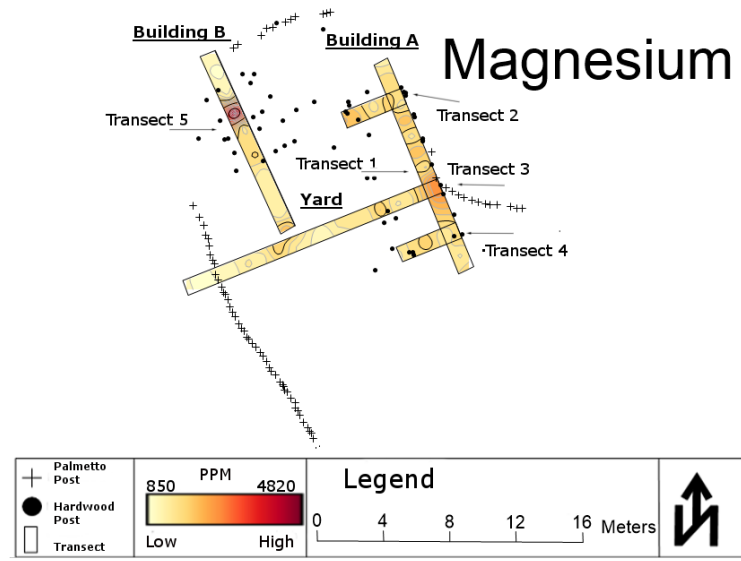


Figure 31. Map of magnesium concentrations.

High levels of manganese appear in the middle of the yard at the end of Transect 5, middle of Transect 3, and middle of building A. Manganese levels fall significantly outside of the site (Figure 32).

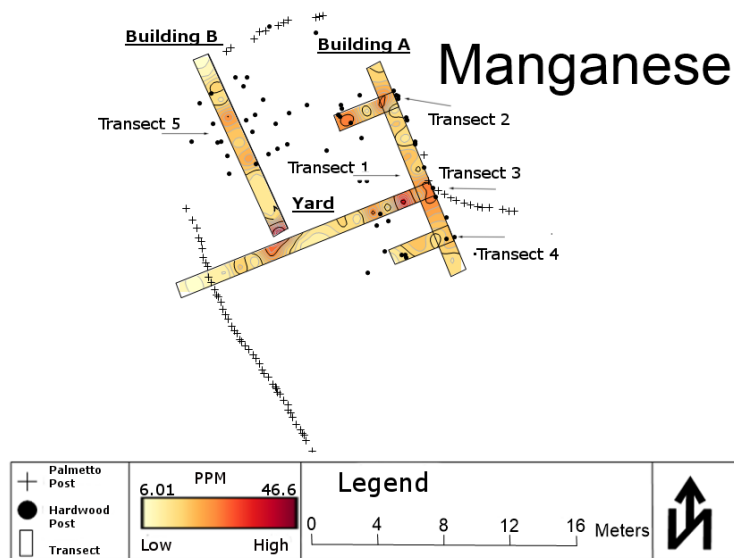


Figure 32. Map of manganese concentrations.



Molybdenum levels are low throughout the east part of Site 74. Levels are high on the west side of the site with medium-high levels in building B and the highest levels in the middle of the yard. Elevated levels extend outside the site (Figure 33).

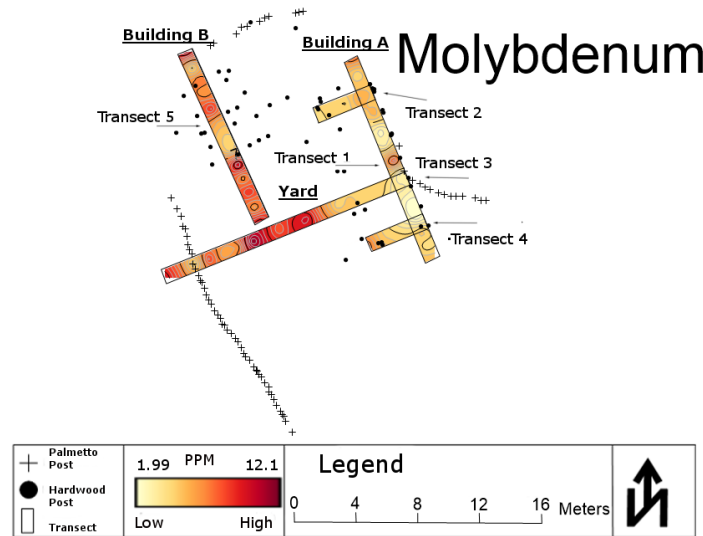


Figure 33. Map of molybdenum concentrations.

Nickel levels are highest in the north east corner of building A and along the walls of building A. Levels are medium-low throughout the rest of the site, with a few spikes in the yard. Levels fall outside the site (Figure 34).

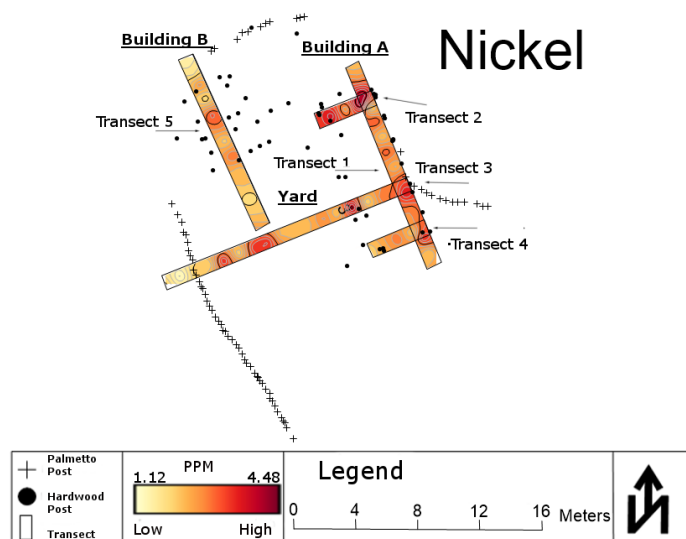


Figure 34. Map of nickel concentrations.

Phosphorus levels are low in building A with a spike along the south west wall.. Medium levels exist in building B, and the center of the yard. Levels fall outside the site (Figure 35).

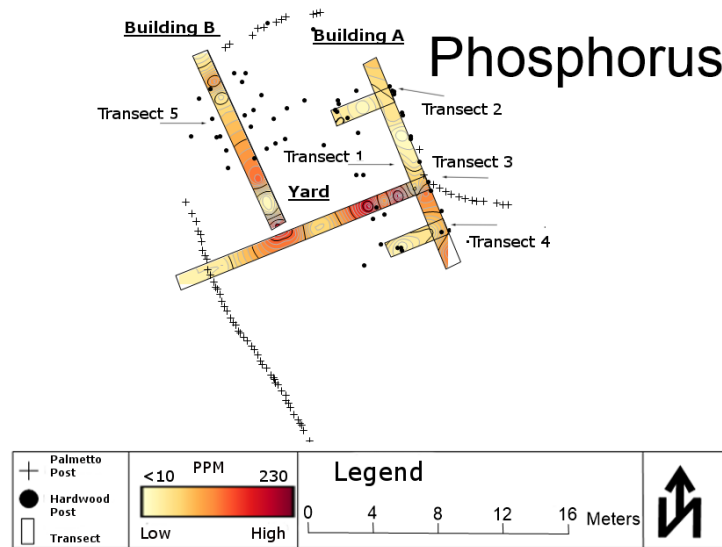


Figure 35. Map of phosphorus concentrations.

Potassium levels are medium-low throughout the site. The highest levels are in building A and the east corner of the yard. Levels drop outside the line of palmetto posts (Figure 36).

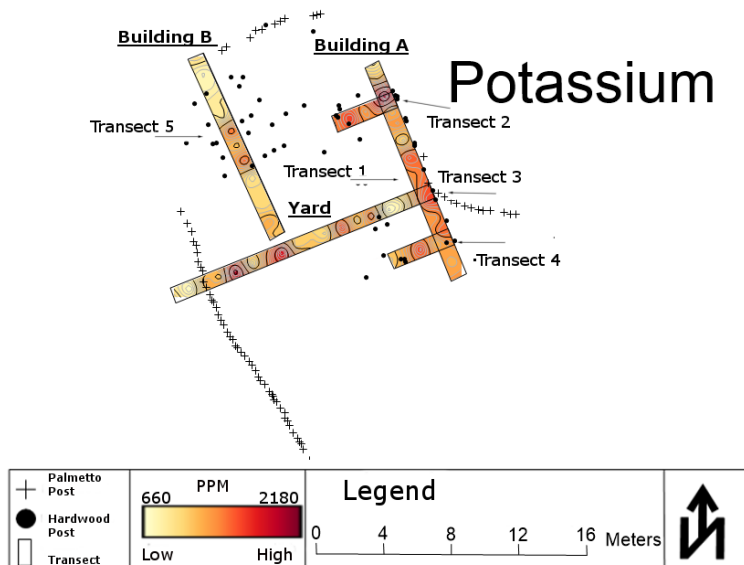


Figure 36. Map of potassium concentrations.

Sodium levels are high throughout the site, but exhibit distinct and extreme levels of variation. Sodium levels are highest in the yard, the south part of building B and along the walls of building A. Levels are low outside the site (Figure 37).

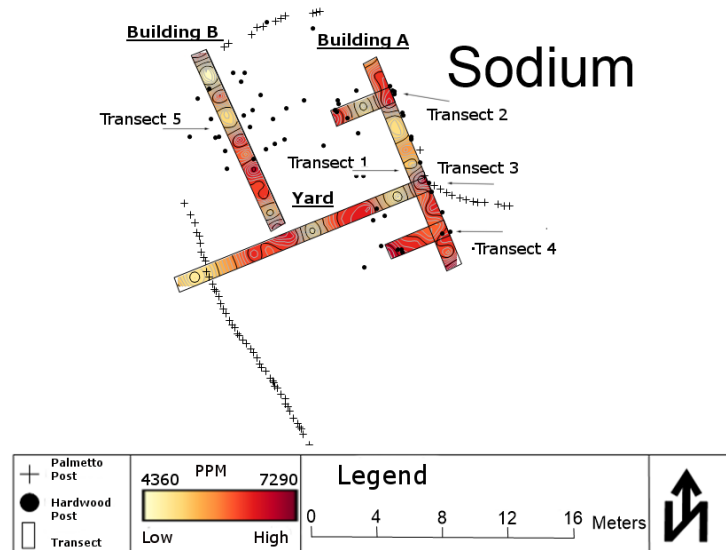


Figure 37. Map of sodium concentrations.

Sulphur levels are low with a medium section near the south east wall of building A and a high spike in the north west of building B (Figure 38).

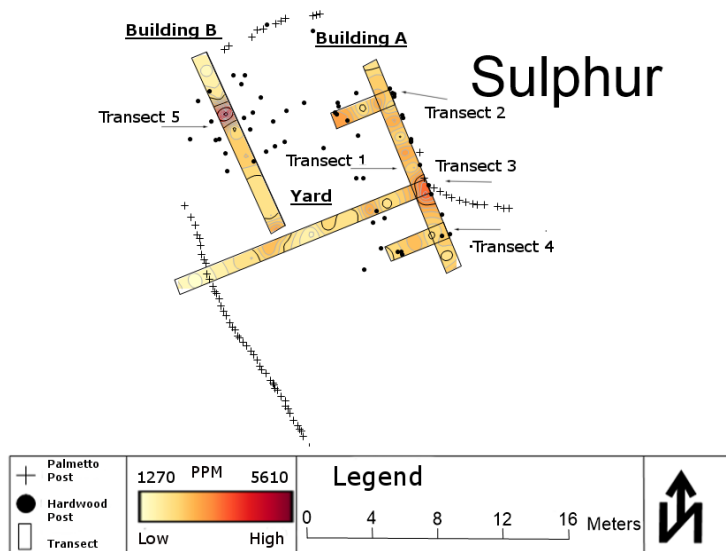


Figure 38. Map of sulphur concentrations.

Zinc levels are low throughout the site but spike along the north wall of building A (Figure 39).

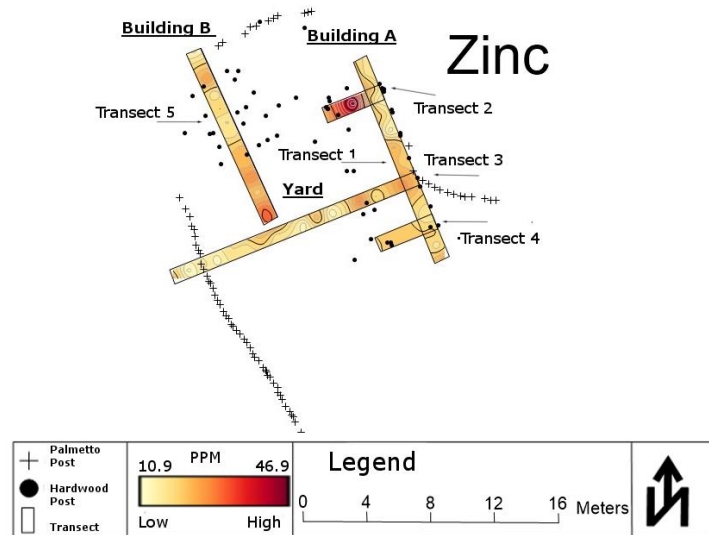


Figure 39. Map of zinc concentrations.

### Loss-on Ignition Results

The amount of organic matter corresponded with the location of the structures. A map of where the loss-on ignition samples were taken can be seen in Figure 40. The highest and second highest amount of organic matter were Transect 3 at meter 16 (39.3%) and Transect 1 at meter 0 (35.5 %). Both samples were outside the site boundary marked by the line of palmetto posts believed to be a retaining wall. The next highest amounts organic matter were both taken from the yard in Transect 5 at meter 12 and Transect 3 at meter 8. The organic matter for each of the yard samples was 33.7% and 30.3% respectively. The organic matter for the samples taken inside the building were lower, half of the %OM from samples taken from outside the site. Inside of building A at Transect 1 meter 7 the %OM was 19.7. Building B at Transect 5 meter 5 was 18 %OM. The average percent organic matter for Site 74 was 29.4%.

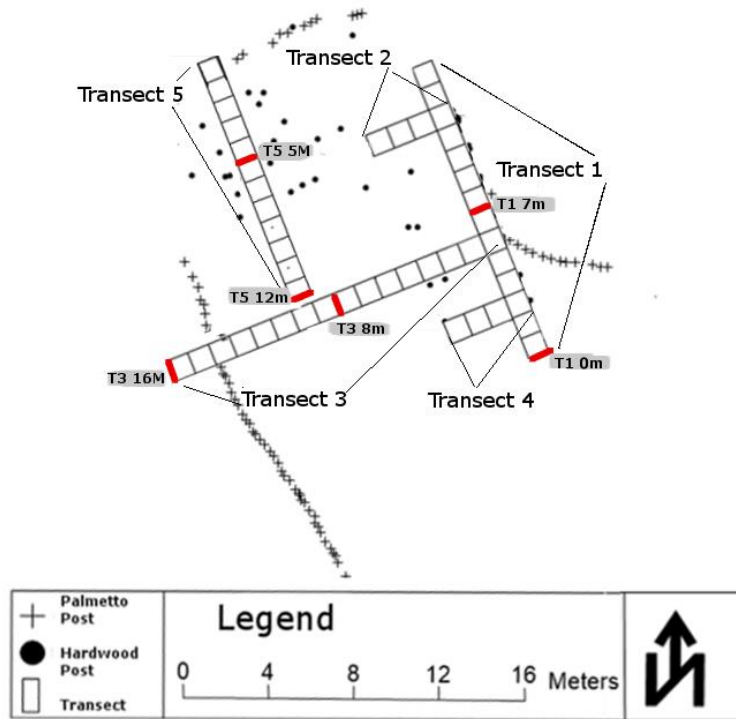


Figure 40. Map showing location of samples taken for loss-on ignition at Site 74.

The column sample was taken from the middle of the yard. The column sample starts at 0cm and was excavated in 10cm levels until a depth of 50cm. Loss-on ignition was performed on the samples. As seen in Figure 41, the amount of organic matter did not increase or decrease monotonically as the depth increased or decreased. Instead, the highest amount of organic matter was in the middle of the column sample at a depth of 20-30cm (40.5%). The other four samples were lower, between 20 and 30%. The amount of organic matter in the sediment from across the site can be seen in Table 3.

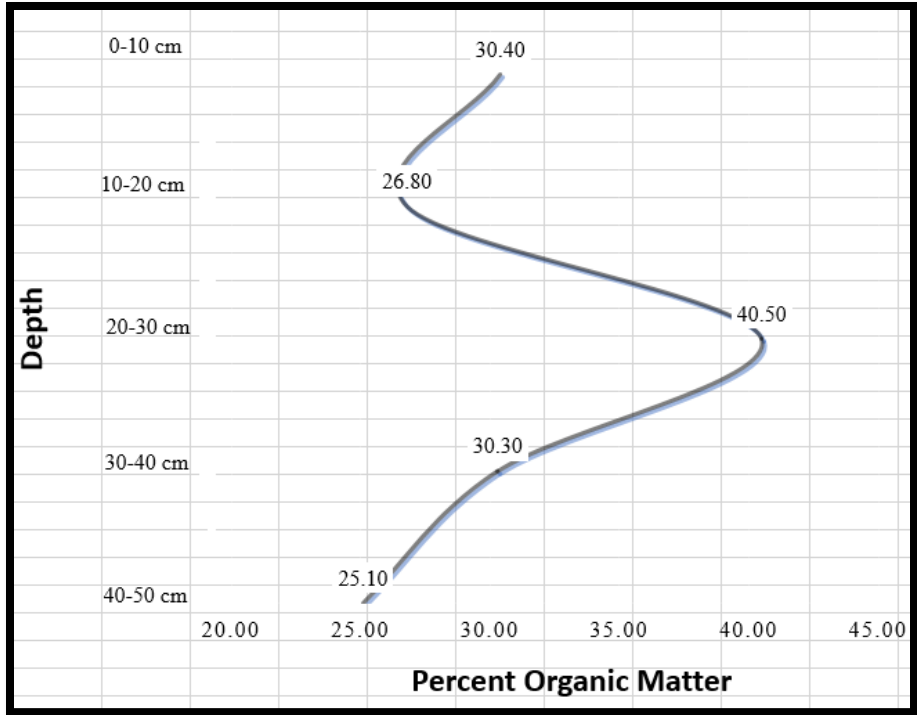


Figure 41. Variation of the Amount of Organic Matter at Site 74 by Depth at Transect 3 Meter 8.

Table 3. Amount of Organic Matter (%OM) in Sediment at Site 74			
Samples From Across Site 74			
TRANSECT	METER		% OM
1	0		35.5
1	7		19.7
3	8		30.3
3	16		39.3
5	5		18.0
5	12		33.7
Column Sample			
TRANSECT	METER	DEPTH	% OM
3	8	0-10	30.4
3	8	10-20	26.8
3	8	20-30	40.5
3	8	30-40	30.3
3	8	40-50	25.1

**Microscopic Sorting Results**

The sediment samples were sorted under the microscope into the five distinct categories: fine red mangrove roots, coarse red mangrove roots, organic, inorganic and unknown. Coarse red mangrove and fine red mangrove roots are sorted separately because fine red mangrove roots may be used for carbon dating. In all the sediment samples, the main component was fine red mangrove roots.

The column sample of Transect 3 at 8m was microscopically sorted (Table 4). All samples from Transect 3 at meter 8 lacked charcoal. The coarse red mangrove and fine red mangrove roots corresponded to each other throughout the samples. The middle sample, at a depth of 20-30cm, had the most roots.

Table 4. Results of Microscopic Analysis at Site 74					
Column Sample					
Transect	Meter	Depth	Fine Red Mangrove Roots (grams)	Coarse Red Mangrove Roots (grams)	Charcoal (absent or present)
3	8	0-10 cm	.22	.09	Absent
3	8	10-20 cm	.15	.04	Absent
3	8	20-30 cm	.37	.08	Absent
3	8	30-40 cm	.32	.08	Absent
3	8	40-50 cm	.23	.17	Absent
Samples From Across Site 74					
1	0	-	.59	.24	Present
1	7	-	.17	.11	Present
3	8	-	.18	.18	Absent
3	16	-	.13	.04	Absent
5	5	-	.31	.05	Present
5	12	-	.59	.24	Present



## Discussion

### Chemical Analysis

The chemical analysis shows five chemically distinct areas in Site 74. These are: the yard, the north section of building A, the south section of building A, building B and outside of the site boundary. The yard shows spikes in sodium, phosphorus, nickel, cobalt, arsenic and molybdenum. Of these elements, arsenic and molybdenum are common elements found soils and sediments and in archaeological soil science have no known ties to human activity. The nickel and cobalt appear in the same area and seem to be related, but without more evidence it is unclear how. Phosphorus, the universal indicator for human activity, is abundant in the yard. Yet, without other artifacts or more chemical evidence, specifics about the cause of the phosphorus cannot be hypothesized. The yard shows clear evidence of human usage, but no specific activities can be discerned from the evidence gathered.

Chemical patterns divide building A into two distinct areas- north and south. The north section of building A is high in sodium and calcium as well as the metallic elements aluminum, chromium, lead, nickel, iron, zinc. These are common elements. However, the variation of their distribution within the site is notable. The variation is most likely because this area was used as a storage area for pots filled with salt and other materials. The pottery was made from clay and tempered with sand, both of which would contain earth metals that leach into the sediment over time. These chemical signatures are similar to the ones found in craft and lithic middens and workshops in the Maya region (Terry et al. 2004; Wells 2004). The south section of building A contains high levels of phosphorus and potassium, which are two elements that indicate burning. The levels of phosphorus and potassium, along with the presence of charcoal, indicate that long-term burning was done in this area, most likely for the evaporation of brine to produce salt. The

same chemical patterns for fires to evaporate brine were also found at the Paynes Creek site of Chan b'i (Sills et al 2016).

Building B has relatively low chemical levels compared to building A and the yard, but has one area in the center of the structure with spikes of the elements boron, cobalt, iron, magnesium, nickel, and sulphur. Of these elements, boron, magnesium and sulphur only appear in high levels here. Though patterns show that these three elements are associated, results are unclear as to how.

On the east wall of building A, at the beginning of Transect 3, and in the center of building B there are a high spikes of sulphur. These sulphur spikes appear in the same area as high levels of potassium and sodium. In the Sacapulas salt makers produced black salt, which contains the mineral apthitalite, that has the chemical signature  $(K,Na)_3Na(SO_4)_2$ . These chemical signatures show it is possible the Maya at Site 74 were producing black salt as well. However, due to the extraction process in ICP-AES analysis which breaks chemical bonds, further research is necessary before that claim can be made.

The low levels of most elements outside of the line of palmetto post indicate that that is where the site ends. Sodium levels, which are high throughout the site, are extremely low just outside the site boundary. The sodium levels may indicate one of two things: little to no human activity occurred here, or the soil here was leached for salt extraction as seen in Sacapulas and hypothesized for the Paynes Creek site of Chan b'i (Reina and Monaghan 1981; Sills et al 2016).

## **Loss-on Ignition**

The loss-on ignition was performed on two sets of data, including one column sample from the yard and six samples from elsewhere across the site. These two data sets overlapped at Transect 3 at 8m and a depth of 0-10cm. Loss-on ignition was performed twice on that sample.

The column sample showed that the amount of organic matter did not vary much by depth, with the highest being Transect 3 at 8m and a depth of 20-30 cm. The highest amount of organic material was located in the center of the column sample. The percent organic matter matches with the results of the microscopic analysis. The samples with the highest amounts of coarse and fine red mangrove roots tended to have the highest percentage of organic material.

Six samples constitute the site sample: two from the yard, two from outside the site boundary, and one each from inside buildings A and B. The pattern of organic matter corresponds directly with location. The highest amount of organic matter was from areas outside of the site boundary, specifically outside of the line of palmetto posts believed to function as a retaining wall. The second highest results were in the yard, and the lowest were from the buildings.

I believe this distribution of organic matter across the site to be a result of artifact distribution. During excavations at Site 74, McKillop and Sills (2016) note that briquetage was most common inside and immediately outside of buildings A and B. Little pottery was excavated from the yard. Over time, the pottery may have broken down into the mangrove peat, adding non-organic material to the sediment and causing lower amounts of organic matter in the areas where high levels of pottery were found.

## **Sediment Composition**

The microscopic analysis was completed on each sediment sample that underwent loss-on ignition. In total, microscopic analysis was done on ten samples at Site 74: Transect 1 at 0 and 7m, Transect 3 at 8 and 16m, Transect 5 at 5 and 12m, Transect 3 at 8m at a depth of 0 - 10 cm, 10 - 20 cm, 20 – 30 cm, 30 – 40 cm and 40-50 cm. The main difference in the analysis is the presence and absence of charcoal. No charcoal is found in the center of the yard. The lack of charcoal is believed to result from burning only occurring inside buildings A and B and not in the yard. The charcoal in the yard is from directly outside buildings A and B and may result from sweeping and cleaning the buildings.

## Conclusion

The microscopic analysis, loss-on ignition and soil chemistry indicate that buildings A and B were enclosed structures with different purposes. The north section of building A appears to have been a storage area for clay and sand-tempered pots filled with salt, as evidenced by the elevated levels of earth metals like iron and aluminum. The south section of building A shows chemical signatures of burning, such as potassium, phosphorus and carbon which appeared in the microscopic analysis of the sediment. The same patterns were found in the south section of building B. I hypothesize that the two buildings were both used for the evaporation of brine to produce salt. The chemical signatures in the yard vary. Although there is phosphorus and potassium, no charcoal was found in those areas of the yard where those elements were found. The areas outside of the wall of palmetto posts was devoid of charcoal and shell and had low chemical levels for most elements tested and a high amount of organic matter. Low element levels may have been due to the lack of human activity in the area or due to the process of leaching the soil for salt as seen in the Sacapulas (Reina and Monaghan 1981; Sills et al. 2016)

How the amounts of organic matter were distributed across the site may indicate where artifacts were located post abandonment. Low amounts of organic matter appear in the buildings where large amounts of pottery were found. The pottery may have degraded, adding inorganic material to the red mangrove peat. In this way, loss-on ignition can be used to reconstruct activity patterns at Site 74 in relation to the spatial use of material items like briquetage. The high levels of organic matter in the yard add more evidence to interpretation that the yard was not used for storage, fires or leaching in the salt-making process.

This research adds to the knowledge of ancient Maya salt works and the peoples who worked there. The study builds upon the methodologies of using chemical analysis to look at

spatial use and determine activity patterns in underwater sites. The sediment chemistry shows that the buildings were used for distinct purposes such as a storage area, a covered area for large fires. The chemistry also shows site boundaries. Hypotheses are supported by the microscopic analysis that shows charcoal only in the areas that chemically correspond to burning. The microscopic analysis shows that the more coarse and fine red mangrove roots are in a sample, the high the percent of organic material a sample has. By studying these “non-permanent” structures, knowledge has been added to the sparse archaeological record about the ancient Maya activities in pole and thatch buildings.

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## **Vita**

Kobi Weaver was raised in southern New Mexico by a wonderful mother and grandparents. She attended New Mexico State University and received a Bachelor's degree in Anthropology in 2012, focusing in Maya Archaeology. During this time she performed research in Belize and Mexico learning archaeological techniques, studying soil chemistry and Maya resilience. After graduating she moved to Louisiana where she taught high school science for two years before continuing her archaeological education under the guidance of Dr. Heather McKillop at Louisiana State University. Here, she worked in the Archaeology and DIVA Laboratory performing 3D scanning, photogrammetry, and curation and preservation of archaeological artifacts. She presented a poster at the South Central Mesoamerican Conference in Tyler, TX in 2016, Tulane University and New Orleans in 2017. She also presented at the Southwest Division of the American Association of Geographers conference in Huntsville, TX in 2017. Kobi was a finalist in the Louisiana State University Graduate School's Three Minute Thesis Competition. She anticipates being a graduate of Louisiana State University and continuing her passion for archaeology, education and activism.