DEVELOPMENT OF A PROTOCOL TO DETECT AND CLASSIFY COLORANTS IN ARCHAEOLOGICAL TEXTILES AND ITS APPLICATION TO SELECTED PREHISTORIC TEXTILES FROM SEIP MOUND IN OHIO

DISSERTATION

Presented in Partial Fulfillment of the Requirements for

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By

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* * * * *

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ABSTRACT

The research goals reported in this dissertation were twofold: to develop a protocol using non-destructive or minimally destructive methods to classify the colorants that were used prehistorically as inorganic/organic and pigment/dye, and to apply the protocol to selected textiles from Seip archaeological site, Ohio. The principle guiding the research was to adapt and sequence the analytical methods permitting the use of the smallest possible sample size which could still yield the most information. Through non-destructive forensic photography prior to any other analysis evidence for the chemical differences on the archaeological textiles resulting from the prehistoric colorant applications were revealed, which facilitated selective and purposive micro-sampling that maximized critical data acquisition while minimizing potential destruction of the artifact.

Pretests on replicated materials were conducted first to assess feasibility and efficacy of selected analytical methods: photography in different lighting conditions (simulated daylight, infrared and ultraviolet), optical and scanning electron microscopy with energy dispersive X-ray analysis (EDS), and inductively coupled mass spectrometry (ICP-MS) for elemental analysis. Differences in chemical signatures on painted replicas, otherwise invisible, were confirmed by forensic photography. While working with replicas, limitations of the analytical methods were discovered and addressed to adapt the methods for the use on
archaeological materials. A specific sequence of modified methods, constituting the ideal protocol, was then applied to selected prehistoric textiles.

Based on the visual examination, eleven textiles from the Hopewellian Seip Mound group were selected and divided into main colored groups: (1) yellow/brown, (2) turquoise/white, and (3) charred. Each of these groups was sampled based on the results of the photography; the turquoise/white group showed patterns otherwise invisible. Optical microscopy illustrated that the yellow/brown textiles were made of dyed rabbit hair with colorant saturated yarns and patterns identical on both sides of the textiles. The two other groups were painted. EDS of the yellow/brown group showed no elemental composition differences between colors, but high organic and copper content as did the turquoise/white group. The charred group showed no significant differences between several colors. However, the red had higher calcium and lower iron concentrations. Two textiles were identified as composite.
DEDICATED

To my husband, Dr. Maximilian O. Baldia for his never-ending support.

To my late great-aunt Itti, Margarete Marxen, for teaching me that learning must never end.

To the memory of my late father, Herman Molitor for passing on his sensitivity.

Stillstand ist Tod.
Im Reiche der Vernunft gibt es keinen.
Wer nicht vorwärts geht, geht zurück;
wer nicht täglich klüger wird, wird täglich dümmer.
Was hilft's, auf seinen Gedanken zu beharren,
wenn um uns herum sich alles verändert.

Johann Wolfgang von Goethe
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CHAPTER 1

INTRODUCTION

“The fabric of a people unlocks their social history. They speak a language which is silent and yet more eloquent than the written word.” Lewis Henry Morgan

Material culture is that part of the world that is modified based on cultural beliefs (Deetz & Deetz 2000). Hence, material culture such as textiles provides information about cultural events and patterns of peoples of the past that otherwise cannot be apprehended. Particularly, adorned textiles function as carriers of cultural meaning, and therefore provide tangible evidence of past lifeways. Often the colors that are used for these decorations play an important role in communicating cultural information as does a white wedding dress in the Western world. The symbolic value of color can be sacred or secular. The Native Americans who were observed by early travelers were no exception. They used color extensively to decorate their bodies or their material objects, both of which had the potential to communicate meanings.

The Seip Mound Group is a Hopewell site located 17 miles southwest of Chillicothe, Ohio, at the center of a large bend of Paint Creek (Appendix A, Figure 1). Burial mounds and extensive earthworks such as those found at the Seip Mound Group sparked the interest of the early settlers and their study started relatively early in the 1800s. Besides the earthworks and mounds, the Hopewell culture in general is
distinguished by the presence of elaborately decorated grave goods found primarily in funerary contexts. Even though regional differences exist, all Hopewell burial mounds contained grave goods that were produced from non-local raw materials such as obsidian, hornstone, copper, and mica. These materials were fashioned into cutouts of animals, celt, headdresses, breastplates, ear spools, and blades. In a few cases, textiles were found at Hopewelian sites as well.

Due to the nature of the textiles, and the limited methods used during the early days of archaeology, the true extent of the Hopewell textile industry can only be approximated and hypothesized. Some textile examples exist, preserved through carbonization, mineralization or due to a peculiarity of the object or the burial environment that kept degradation at a minimum. Even then, archaeological textiles may not be representative of the prehistoric textile industry, especially with respect to coloration. However, the study of the surviving textiles constitutes a starting point to understand Hopewelian textile production and material preferences.

Typically, textiles degrade in burial contexts, leaving them brown or black in appearance. While these textiles do not necessarily appear to be colored with dyes or pigments, it cannot be assumed that fabrics of the past were not dyed or pigmented in some manner to decorate them. Among other artifacts, over 200 textile fragments of varying size and degree of degradation were recovered from the Seip Mound Group. Some of these textile fragments still exhibit colored patterns, but most do not.

Willoughby (1938:273-287) documented some of the fabrics that were uncovered at the Seip Mound Group (Appendix A, Figure 2, Textile Structures as drawn by Willoughby. A drawing of the colored pieces is represented in his figure 2 (276), but also
his illustration (his figure 1) labeled “o” (275) is described as a “fragment of a mantle in close twined weaving preserved by contact with copper plates. The background is a dark maroon with designs in clear yellow, outlined in black. The colors produced by dyeing or painting. From a multiple burial, Seip Mound, Ross County (276).”

Furthermore, he describes this piece as “a fragment of a sash or loin cloth and the small piece of which the drawing was made has a circular spot of reddish brown color which seems to indicate that the cloth was originally decorated with a design in one or more colors by staining” (277-278). Another textile from this mound consisted of vegetal fibers that had been neatly wrapped with rabbit hair that is “twisted…and stained red…the preserved portion of the fabric had lain in contact with some copper objects” (278). In addition, he describes the buried bodies as being covered by “one or more large mantles or shawls which lay in contact with three copper plates of the type found in many mound burials. The cloth where it came in contact with the copper was remarkably well preserved when first exposed to the air and showed very clearly the stained color design (emphasis mine) with which the garment was decorated (281)…Fortunately the preserved portion of the designs were copied in water colors…before the drying and checking the cloth adhering to the copper had practically destroyed both design and coloring….The background is a dark maroon, the designs are in clear yellow outlined in black and are shown about one-fourth of the natural size…” Willoughby further suggests that it would be difficult to reconstruct the design from the small textile fragments. He has however observed similarities of these designs to the “incised drawings upon bone, stone and other materials shown in my paper on art of this people (282)”. He also describes a bag found at Seip that has colored stripes consisting of “two warp cords dyed brown, separated by a
single undyed cord (286)” as being similar to the bags that the contemporary Ojibwa or their neighboring tribes used.

Although some researchers have already studied the Seip textiles since Willoughby’s description in 1938 (Church 1983, 1984; Song 1991; Thompson 2003), colorant use has thus far only been described, but has not been considered for further studies.

Color can be achieved by the use of organic or inorganic colorants, both requiring significantly different techniques due to their physical properties, but also leaving specific chemical signatures behind. North America has a long history of colorant use as is exemplified by the use of the inorganic mineral pigment red ochre, which was used from the Clovis through the Mississippian periods in domestic and mortuary contexts. Inorganic colorants are comparatively easy to analyze because they are of mineral source. They may change their chemical species, but not their elements through time. On the other hand, organic colorants from plant or animal source such as dyes are far more difficult to analyze, because they decompose and leave more obstructed direct evidence if any. Furthermore, determining the use of organic colorants is a complex issue. For example, a plant can produce several colorants, and a single colorant can be obtained from several plants. Therefore, organic colorant sources have received far less attention in eastern North America than inorganic colorants have.

Colorant use and importance was first witnessed by early travelers and explorers and later corroborated by ethnographic reports. According to the accounts of travelers to the new continent in 1607, the settlers learned to use native plants such as puccoon as dye from the Indians who used plants to dye their cloth and for body painting (Smith 1986
In the 1830s Catlin described that native groups in eastern North America wore colorful clothes, used body paint for adornment and carried medicine bundles that were dyed in different colors: black, white and red (Catlin 1995).

According to Kehoe (1992), color is noted as a means of communicating status and prestige, clan affiliation and intentions of war. For example, the Muskogeans and the Cherokees in the eastern Woodlands subscribed to the “white and red paths” where the white-path-people argued for peace, while the red-path-people were expected to argue for war during times of decision making (Kehoe 1992:184).

Lewis Henry Morgan (1954 [1851]) describes the importance and use of colorant by the Iroquois:

“… Near the rump of the moose and near the neck … there are small tufts of white hair about four inches in length, each yielding a small handful. These hair were carefully preserved, dyed red, blue and yellow, and used in the manufacture of the finest varieties of burden straps…(19); [furthermore]… sometimes they [wampum] are all in one color, in others variegated, and still in others woven with the figures of men to symbolize, by their attitudes, the objects or events they are designed to commemorate … (54).”

Later ethnographic studies of the Indian groups in eastern North America note native plant knowledge and dyeing of fabrics with locally obtained plant materials. Densmore, for example, provides extensive lists of dye plants used and, in some cases, dyeing recipes (Densmore 1974).

While archaeological, ethnographic, and historical records indicate that color was important, and that pigments and dyes were used extensively, actual color analysis from eastern North American archaeological textiles has only rarely been conducted. Therefore, the extent of colorant use in prehistory is likely to have been underestimated.
Problem Statement

Ethnographic and historical evidence supports the notion that some prehistoric textiles were colored either with pigments or dyes. However, direct evidence of coloration on the actual artifacts is not readily visible in most cases due to degradation, charring, mineralization or any other alterations in the burial context. If the color is, in fact, visible, neither the colorants nor the method of colorant application have been identified. This however limits our knowledge about prehistoric exploitation of the environment or of possible trade for these materials. Therefore, detection of colorants on textiles is an essential first step that could lead to new insights into prehistoric cultures.

The development of a protocol to visualize coloration present in archaeological textiles and then purposely sample representative areas, followed by a sequence of analysis that cumulatively yields data is essential to study coloration on the few textiles that have survived to this day.

Consequently, the research objectives are:

- Develop prototypes that simulate the expected properties of the colored textiles from the Seip Mound Group by applying different colorants to fibers similar to those described by early travelers and in historical and ethnographic records.

- Use these prototypes to evaluate analytical methods (pp. 32-41) of forensic photography, optical microscopy (OM), scanning electron microscopy and energy dispersive spectroscopy (SEM-EDS), inductively coupled plasma mass spectrometry (ICP-MS), and inductively coupled plasma optical emission spectrometry (ICP-OES).
• Refine methods as needed and determine an ideal protocol which delineates a sequence of methods that provides the most data with the smallest sample size.

• Apply the determined ideal protocol to selected textiles from the Seip Mound Group comparing colored and non-colored areas.

• Refine the protocol based on the outcome of examinations of the actual archaeological textiles comparing colored and non-colored areas.

• Explore results of the protocol application to the selected Seip textiles for further use in archaeological textile research.

• Explore the results of the protocol application to the selected Seip textiles with respect to our knowledge about the Hopewell peoples.

_Justification_

Most tests for the presence of colorants such as chromatography require sample destruction; multiple samples will eventually destroy the artifacts. Thus, these methods are undesirable even though they yield valuable information about colorant constituents. Other more desirable approaches need to be explored, or at least used as a starting point before destructive analysis of archaeological textiles. A sequence of methods should start with those that reveal visual evidence for coloration, followed by microscopy and EDS of purposive micro-samples whose sampling is informed by the visual evidence. Thereby, the most data can be gained for the least amount of sample; hence, destructive treatments of the already limited archaeological textile material would be kept at a minimum. Therefore, non-destructive methods such as forensic photography under UV and IR lighting conditions were employed to detect possible patterns where colorants were
applied. These show selective absorption, reflectance and fluorescence on the textile materials in regions of different colorant chemistry. This visual evidence facilitates the selective sampling of micro-amounts of material that can still yield critical data.

Representation of variation in the composition of archaeological textiles is difficult. Rather than randomly sampling archaeological textiles, selective sampling informed by IR, UV fluorescence and reflected photographic methods minimizes potential textile damage while maximizing the information provided by further chemical analysis of these samples. Subsequent analytical methods such as mass spectrometry must be adjusted to deal with reduced sample sizes and the potential contaminations of a typical archaeological material.

With respect to the Seip textiles, the interpretation of information about the Hopewell gleaned from a variety of artifacts is incomplete if textile analysis but especially colorant analysis, is not included. In comparing the patterned and colored textiles with the apparently non-patterned, non-colored textiles the suitability of the protocol could be tested and inferences about prehistoric artisanship and resource management could be made.

**Assumptions and Limitations**

The features of purposive sampling could be considered a form of a systematic, stratified sample acquisition, whereby the chemical signatures of the textile materials define the stratum of the coloration possibilities, each of which is sampled. However, for the purpose of this research, selective, purposive sampling should not be seen as a statistical exercise and is not treated as such. Instead, it should be considered as a focused means of acquiring qualitative data from a very limited population that has survived and
should not be destroyed for the sake of gaining data. The statistical implications must be addressed elsewhere. Therefore, it is assumed that the surviving textiles are representative of the textiles that were produced and consumed during the Hopewell heyday. Furthermore, it is assumed that the selected textiles that were sampled are representative of the prehistoric Hopewell textile population used in mortuary context.

The Seip textiles that were analyzed were recovered from the same site but no information is available indicating the particular mound where any of these were found. It is assumed that these (1) Seip textiles were exposed to the same formation processes, and hence, should degrade in the same fashion unless there are differences in the way the textiles were altered prehistorically. (2) The Seip textiles are contemporaneous. (3) The colorants still exist in adequate quantity on the fabrics so they can be detected and recognized as such. Furthermore, if heavy elements such as iron are detected, (4) it is possible to distinguish contaminants from colorants that were applied prehistorically.

This research is limited to the colored and non-colored textiles excavated from Seip Mound Group in Ross county Ohio that are curated at the Ohio Historical Society in Columbus, Ohio. The Seip Mound group is considered Middle Woodland (C14 Dates), which restricts the results of the study proposed herein to this time period. The number of available textile fragments is limited, and these fragments do not reflect the full range of textiles that were produced by the Hopewell peoples. Another limitation is the lack of exact provenience. Few of the Hopewell textiles have information beyond a reference to the mound or mound group, and even that is often missing. The Seip textiles are no exception. However, Greber (1983) notes that the mounds should be considered complexes of sites, particularly Seip and Harness, and that these were constructed and
used over substantial time spans (Konigsberg 1985, Greber 1997); according to Thompson (2003) this historical depth makes it even more important to focus attention on technological changes over time.

The textile fragments examined have been stored between glass plates, often several in the same case. However, it is not necessarily known if those fragments that are housed together indeed belonged to the same textile, and no records exist indicating reasoning for choosing to put particular fragments together into the same housing. Furthermore, only the individual glass plates have an accession number, while a different number does not identify the fragments within each pair of plates. Thompson (2003) developed an additional numbering system, by which some of these materials were labeled in her dissertation. Her protocol was combined with that from Song (1995) in this research to provide continuity in research in the future.
CHAPTER 2

LITERATURE REVIEW

This chapter covers a review of literature of those areas relevant for this research:
The first section covers archaeology related to the Hopewell culture in general and the Seip Mound Group in particular, past research on Seip textiles and research on North American archaeological textiles with respect to coloration. In the second section, colorants and their sources are briefly discussed, and the differences between dyes and pigments and between organic and inorganic colorants are delineated. In the final section, background information about the chosen analytical methods is summarized.

Past Archaeological Research

The research on the Hopewell mounds started early in the 19th century, culminating in a large body of literature. Only the most relevant writings about the Hopewell in general are reviewed here with particular attention to the textiles that were recovered in Seip mound.

The Hopewell

The Early Woodland Adena culture preceded the Hopewell cultural group (Appendix B, Hopewell and Adena C14 Dates). The Adena peoples already practiced an
elaborate burial ritual that is characterized by log tombs, generally circular charnel houses, cremation, and the use of red ochre pigments. Some exotic grave goods were already found in Adena burials such as gorgets and engraved tablets with traces of pigments adhering to their surface (Appendix A, Figure 3, Adena Tablet). The latter have been interpreted as stamps for decorating clothes and body (Jennings 1989). Thus, the predecessors of the Hopewell left evidence of colorant use and a specific manner of applying them.

Conical burial mounds, vast earthworks and enclosures centering at a 150-mile radius around Chillicothe, Ohio, but also extending into Indiana, Kentucky, West Virginia and Pennsylvania, characterize the Hopewell culture. The Hopewellian were, according to Fitting (1978:45), the “most spectacular and still best known cultural manifestation of the Middle Woodland time period”. The geographical distribution of archaeological sites associated with the Hopewell reaches from western New York to Kansas City and from the Gulf of Mexico to Lake Huron (Appendix A, Figure 4, Geographical Distribution of Mound Sites).

The traits that all Hopewell sites have in common include “burial mounds often occurring in groups of several, and earthworks; distinctive dentate-stamped and rocker-stamped ceramic vessels, often with cross-hatched rim ornamentation and zoned decorations; platform pipes, sometimes carved with animal and bird effigies; cut animal jaws and teeth; pan pipes…with regional manifestations that are distinctive…in spite of widespread similarities…(Fitting 1978:45)”, (Prufer 1964a; Struever and Howard 1972; Fitting 1978, Woodward and McDonald 1986), (Appendix A, Figure 5, 6 and 7 Hopewell Artifacts).
These similarities led Prufer (1964b) to postulate a religious cult or at least a relationship between communities based on shared values, which Caldwell (1964) named the Interaction Sphere. While many of the artifacts that are associated with the Hopewell were found in both burial and habitation sites (Struever and Howard 1972:49), the textiles have been recovered from burial mounds only (Ericksen and Jakes 1997, Mills 1907, 1909, 1916; Morehead 1896; Shetrone 1926; Shetrone and Greenman 1931; Webb and Snow 1974, White 1987; Willoughby 1938). Another identifying feature of the Hopewell is the evidence of a widespread exchange system, in use since the Archaic period, and reaching its peak during the Hopewellian period.

Gulf Coast conch shells found their way to the Great Lakes, while copper from Lake Superior was traded to the southern regions of the U.S.. Grizzly bear teeth and obsidian from the Rocky Mountains were found in Illinois and Ohio, while the Ohio tool stone from Flint Ridge was found in burial contexts far away from Ohio (Fitting 1978:45).

Mortuary practices of the Hopewell varied greatly. Fitting (1978) summarizes that the western Hopewell mounds contained inhumations, usually in log tombs, but also secondary burials were found in many of these mounds. In comparison to Illinois, the Ohio Hopewell left behind larger mounds containing more elaborate cremation burials as well as inhumations, with more variation and complexity. The size of the mounds and associated earthworks is so extensive that repeated use of these sites for burials over many generations is suggested. The chronology differs regionally.
Seip Mound Archaeology and History of Research

The Ohio Hopewell sites can be divided into four categories: the early Hopewell (Tremper, some of the Mound City mound group), middle Hopewell (Seip, Harness, Hopewell and Rockhold), late Hopewell (Turner, Marriot-1, McGraw, Brown’s Bottom, Ginther, Newark and Marietta) and the latest Hopewell, which is represented by the hilltop enclosures in the southern part of Ohio (Prufer 1968:148). For this research, the focus was on the Seip Mound group (link Appendix B, C^{14} Dates).

The Seip Mound Group is located 17 miles southwest of Chillicothe, Ohio, at the center of a large bend of Paint Creek (Appendix A, Figure 8, Squier and Davis Map). It is a large, geometric earthwork consisting of two circles and a square as perimeter with three conjoined large mounds and several small mounds in the center. Atwater first described this mound group in 1820 (Mills 1909). Squier and Davis provided a map and a description in 1848. They described the Seip complex as follows:

Within the large circle, and not far from its center, is a large elliptical mound, two hundred and forty feet long by one hundred and sixty broad, and thirty in height. It is considerably larger than any other single mound in the valley, and covers a little more than two-thirds on an acre. …to the right of this fine mound is a group of three others in combination.…

The large elliptical central mound, noted as figure ‘a’ in the Squier and Davis map, was first called Pricer Mound after the owner of the land. The Seip Group referred to the three conjoined mounds (figure ‘b’ on the map) (Mills 1909; Shetrone and Greenman 1931) and only the conjoined mounds were excavated in the 1906-8 field seasons by Mills. The second major excavation of Seip was conducted by Shetrone and Greenman for the Ohio Historical Society (OHS) from 1925-1928. This time the elliptical mound was excavated, and Shetrone and Greenman called this mound Mound Number
One (Pricer in 1906). The three conjoined mounds that had been called Seip Group by Mills were renamed Seip Mound Number Two. This renaming of the mounds and the change of field supervisors resulted in much confusion in the cataloguing of the artifacts at OHS, exacerbated by the fact that frequently provenience of the recovered artifacts was not noted (Greber 1976). Furthermore, the field notes from the 1906-8 field seasons were lost. The only results available are those in published reports by Mills, which do not note provenience.

According to Shetrone and Greenman (1931), the Seip burials exhibited evidence of both cremation and in-flesh burials. They reported that “…the great majority of the burials in Mound number 1 were cremated and lay upon specially prepared earthen platforms built up a few inches above the floor…[that was ] made of clay, gravel or sand with coverings of charcoal…, which suggests that the cremation was accomplished not far from the burial site…”, but Shetrone and Greenman also suggest that the “…charcoal was not in every case a result from the cremation; there was charcoal found…and the remains were not cremated… (1931:480)”. They also report that a thin layer of bark covered most of the burials, but they are unsure whether this bark actually covered the dead or if it was the remainder of a bark roof that had collapsed. A total of 129 burials were found in Mound Number One with only four not on a platform. There also were eleven intrusive burials. The burials on the platforms were either cremated, in-flesh or partially cremated; 69 were single graves and the others had two or more individuals buried together. The most prominent burial, the “great multiple burial (1931:369)”, contained six in-flesh interments (four adults and two children). The burial chamber was built of “logs placed above one another and secured by large stones (1931:370)”. This
functioned as a burial chamber to protect the burials from the pressure of the earth. Over this burial chamber a textile canopy was placed and secured by over a hundred bone skewers. The burials were accompanied by a large number of grave goods such as numerous pearls, copper breastplates and buttons, and mica. Shetrone and Greenman (1931) also reported that they found “… a portion of fabric or burial shroud bearing a design in color… (376)” beneath a breastplate. At Seip Mound the dead were also placed in charnel houses (Mills 1909). The largest combination of burials contained four individuals in a single cist. Mills illustrated that:

“frequently the floor around the posts would be covered with great quantities of charred cloth, ornaments, and implements, … It seems very probable that the clusters of posts near the graves were the sacred shrines for the dead, and here the clothing, and very frequently, some of the most interesting ornaments [were found]… in a few instances, copper ornaments were found with the charred woven fabrics, so promiscuously placed upon the floor surrounding the posts…(1909:286)”.

Mills (1909), and Shetrone and Greenman (1931) agreed that the Seip population practiced cremation after the bodies and associated artifacts had been covered by textiles, which led Song (1991) to conclude that the charred textiles curated in the OHS were likely those textiles that had been found associated with the cremation remains. On the other hand, none of the textiles that Shetrone and Greenman (1931) described were labeled as charred, blackened or carbonized. They did however suggest that some of the textiles had been (1) preserved due to contact with copper, (2) pieces of the canopy, (3) pieces of a burial shroud without association to copper, and had been (4) used to cover a burial. They also identified five different construction techniques. They suggested that the fabrics with colored designs were found in association with seven copper plates, and that these displayed “characteristic Hopewell design”. They gave details that
“...The incompleteness of these two patterns seems to indicate that the breastplates were originally fastened to a larger piece of fabric which was painted the entire pattern. The background is maroon, with the tan design outlined in black. The designs are not the result of weaving colored threads into the fabric but were affected by means of staining or dyeing with mineral pigments, possibly by the use of stamps. The print of these designs was transferred to the copper plates, on which they are still to be seen clearly... (451-452).

The fabric canopy over the “great multiple burial” was made of a “...simple open weave...[of the] thin quality of modern burlap (452)”. Furthermore, Shetrone and Greenman (1931) suggest that the materials used to make the “fine twisted strands ...were probably taken from the inner bark of a tree...[and that the] twilled pattern of the matting [was] done in split reeds (455)”. They proposed that the fine material with the “characteristic Hopewell designs” adhering to the copper plates were made of swamp milkweed (*Asclepias incarnata* L.).

In the cremation process that took place at Seip Mound Two in which the body was burned with coverings of straw, twigs and fabric, Mills (1909) suggested that the materials that were used to produce this cloth must have come from trees and plants.

Seip yielded the largest number of textiles recovered from any Hopewellian burial mound in Ohio, comprising about 50% of the total textiles recovered from 10 sites (Hinkle 1984). Willoughby (1938) studied and described the Hopewell textiles extensively, classifying them based on fabric structure. He asserted that none of the textiles showed evidence of manufacture on a loom (273). He also suggested that some of the materials that were used as fiber sources were plants such as Indian hemp (*Apocynum cannabinum* L.), nettle (*Urtica sp.* L.) and the inner bark of linden (*Tilia americana* L.), slippery elm (*Ulmus rubra* Muhl), and other trees. More structural studies were conducted by Church (1983), Hinkle (1984), Thompson (2003), White (1987) and
Wilson (1979) of whom Thompson specifically focused on the charred materials.

Textile Research with Respect to Coloration

The focus of most research on archaeological textiles of North America has been fabric structure with detailed descriptions of appearance. Church (1983) concentrated on the structure of Seip textiles curated at the Ohio Historical Society, claiming that some of the textiles are “painted” and some of these were even further decorated with shell, pearls, copper, and mica, thereby requiring a tremendous amount of energy to produce. She proposed that the decorations on the “painted” textiles and their associations with the burials were meant to communicate social identities or roles. However, she did not pursue the study of the colorants themselves.

Song (1991, 1996) examined fibers from selected Seip textiles that are curated at the Ohio Historical Society, and found that specific structures were correlated to specific fiber contents. She analyzed fibers from those fragments that retain obvious coloration and she found the textiles that were “painted” were constructed of animal hair. She offered three possible inferences. First, the textile may have been painted to alter the natural color. Second, it could have added symbolic value, especially since the painted motifs were of the design recognized by Shetrone and Greenman (1931) to be typically Hopewell. Third, the rarity of the animal fiber composition could be linked to further uniqueness. She concluded that the designs may have had symbolic value. However, she did not study the coloring materials themselves.

Willoughby described the Seip textiles in detail, but also recognized that exposure and the handling of the specimen “practically destroyed both design and coloring… [that
had been] a dark maroon, the designs…clear yellow outlined in black…”. Furthermore, he suggested that particular techniques such as painting or staining were applied to create the colored patterns. However, he did not describe all of the Seip textiles (Willoughby 1938:273-287).

The use of color by prehistoric peoples was also addressed in Willoughby’s discussion of textiles found at the Mississippian temple mound sites of Etowah, Georgia and Spiro, Oklahoma. While these sites existed a thousand years later than the Hopewell, they provide evidence of coloration of fabrics prior to European contact. The Etowah textile remains were covered with motifs of concentric circles enclosing an equal-armed cross arranged alternately with a larger cross without rings and having a central disc. The natural light color of these designs appears against a dark red background (Willoughby 1938:279). Examining these materials today, one may see patterns in shades of black (Kathryn A. Jakes, personal communication 2003), and the colors as Willoughby saw them at the time of the excavation are no longer visible, which makes these early descriptions even more important.

Spiro Mound in Oklahoma yielded textiles that “displayed flag-like figures of red and black … composed of a mixture of vegetal fiber and rabbit hair … dyed either red or black that were produced from yarn-dyed materials” (Willoughby 1938). These colored textiles range in color from rose red to gray (King & Gardner 1981) and may have retained their color because they were recovered from a dry climatic environment. Kuttruff proposed that the complexity of the textiles including the colored patterning and design motifs is indicative of sophisticated artisanship and status differences that were expressed through clothing (Kuttruff 1993), but her study did not involve the
identification of colorant types. Saltzman (1963) identified madder as the dye plant that had been used to color some of the Spiro textiles, but he was unable to determine the species.

Martoglio et al. (1990) and Jakes et al. (1992) used microspectroscopy to find evidence of dyes, and were successful in applying this method when working with South American textile samples. However, when applying this technique to a single small sample from an Etowah textile, the results were inconclusive due to the smallness of the sample that the researchers were able to obtain. The authors noted additional limitations in the infrared indication of colorants on archaeological fibers, including the lack of a comparative dye plant collection and associated spectra.

Sibley and Jakes (1992, 1994) researched Etowah burial 57 textiles. One of these displayed red, gold and dark brown colors. Since it contained no iron, the iron-containing minerals that are thought of as ochre could not have been the source of the colorant. Furthermore, the researchers found organic bands in infrared spectra of these fibers that revealed not only the proteinaceous nature of the mineralized fibers, but also other bands that were attributed to dye.

**Colorants**

The study of colorants that can be applied to textiles is inherently complex, because it cuts across several disciplines and traditions: geology, botany, dye chemistry, material analysis, textile science, art, history, and prehistory. When dealing with archaeological specimens, the process is even more complicated because materials are likely to have been altered due to their environment during formation, subsequent
degradation, and handling during and after excavation and in curation. Furthermore, dyes and pigments dictate the methods by which they can be applied to produce the desired colors. All colorants produce color as a result of their chemical structure, i.e. by the presence of conjugated double bonds and associated functional groups. These systems are called chromophores for their ability to absorb visible light and to reflect some of that energy, which is then perceived by humans as color.

Colorants can be classified by several different means, which potentially creates confusion since the nomenclature is not applied consistently. The size of the colorant molecules determines whether or not a colorant is considered a dye or a pigment, which essentially changes the way the colorant can or must be applied and how it interacts with the substrate. Furthermore, the chemical composition, be it organic or inorganic must be considered, because this also affects the properties of the colorants. Those colorants that have been identified in archaeological or historical context and the implications that their physical properties dictate are briefly delineated in this section.

**Pigment or Dye, Organic or Inorganic?**

Color for dress or body adornment purposes can be achieved by the use of either pigments or dyestuff. Physically, the dye and pigment differ in molecular size, pigments being much larger than dye molecules. Furthermore, natural pigments generally are inorganic, and they adhere to the surface of the fiber aided by a binder or glue. This could be anything to make the pigment stick to the substrate. Historically, such media as egg white, water, clays, oils and resins have been used as pigment binders.
By contrast, a dye molecule interacts chemically with the substrate at a molecular level. Chemical bonds or associations are formed, thus best results are achieved when the two components are compatible. If the substrate/dye combination is ideal, a more durable, fast and level color is created. Dyes can be applied at any time during the textile production process, while the pigments generally are applied to a finished textile. Pigments can also rub off more easily, because they are not chemically bound to the substrate.

An organic dye that has already entered fibers at a molecular level can form a much larger complex molecule, sometimes called a lake, in the presence of a metal ion and a chelating agent. In this process called mordanting, this complex acts as chelate between dye and fibers creating much faster colors because the now larger molecule is confined to the inside of the fibers. Sometimes this lake is also labeled as pigment even though the source of color is organic.

This label as *organic pigment* describes the large molecule it forms but it is also potentially confusing, because the colorant is located within the fiber. According to Berns (2000:132), the “greatest validity” to differentiate between pigments and dyes is the presence or absence of the binder, which makes the pigment adhere to the fiber surface.

Pigments from inorganic (mineral) sources have been found in many North American archaeological sites, suggesting plentiful use. For instance, Brown (1976, 464) lists a variety of minerals that were associated with Spiro as follows: ochre as source for red and yellow, glauconite for green, manganese ore for black and purple, and galena and kaolin as a source for gray and white. The Haley site yielded limonite which is a source for yellow and cinnabar which is a source for green (Boyd, 1967).
However, some of the colors found in archaeological remains such as the textiles cannot totally be explained by the use of the inorganic pigments alone. If pigments were used, elemental analysis should disclose the presence of metal ions.

The colors in the Spiro textiles include a rose red, pink, yellow, grey, tan, brown-black. King and Gardner (1981) claim that they were colored by some sort of resist dye methodology. Because pigments must have a binder to “paint” or coat fiber surfaces they could not have been the source for these colors if a resist technique was in fact the method of Spiro textile coloration.

Furthermore, the types of yarn structures that Willoughby (1938:273-287) described in the Seip textiles as “vegetal core with red-stained rabbit hair wrapped around it” can only be achieved in coloring the fibers before spinning. Therefore, the prehistoric artisans had to have specific knowledge about dyes and how to apply them.

While the identification of inorganic pigments is relatively straightforward and their sources can be recognized through comparison of trace elemental composition, the organic remains from dyes are much more difficult to detect and interpret in an archaeological assemblage since organic materials degrade readily. However, some evidence might be gained by looking for plant evidence of dyeing as was shown by Tomlinson (1985) who used botanical remains to identify dye plants from a 9th century A.D. waterlogged deposit in Britain, and Hall (1996), who surveyed paleobotanical evidence for dyeing and mordanting.

Furthermore, since plants that were probably the most common source for dyes in the past also could have had other uses such as food or medicine, the presence of plant remains in archaeological contexts does not necessarily support their use as dyes. For
example, walnuts are food but their hulls yield a good black or brown colorant (Casselman 1993). Moerman (2000) identified sumac (2000:473) and bloodroot as medicine (2000:515), but also suggested their use as dyestuffs. Ethnobotanists have labeled seed assemblages as evidence of prehistoric diet, but some might also be related to coloration (Jakes and Ericksen 2001). These multipurpose plants make interpretation difficult and justify further material analysis of the archaeological textile remains themselves rather than focusing on the correlates.

**Known Inorganic Mineral Pigments**

The most prominent pigments that were used prehistorically are the iron oxide pigments, often referred to as “ochre”. While the term is used widely, it is also problematic because in the strictest sense it only denotes the color it produces, even though it is often used interchangeably with hematite (Fe₂O₃). In fact, the term “ochre” does not describe the elemental composition, which can vary greatly, and in some cases, the ochre material described does not necessarily even contain any iron. [Note: The terms ochre and hematite are used herein as they have been used in the referenced sources. Both terms are used to describe color producing minerals and they are defined in detail in the glossary section].

“Ochre is suggestive of ritual activity, especially in human mortuary contexts, but it is also a preservative and an abrasive material and also may have had functional uses. It is regularly found in varying amounts in Paleo-Indian sites of all ages. All items are heavily encrusted with red ochre, suggesting a ritual significance” (Frison 1998).
Examples of ochre use in mortuary contexts wherein hematite covered bones and associated tools were found include the Anzick site, Montana (Lahren and Bonnichsen 1974) and the Gordon Creek burial site, northern Colorado (Anderson 1966). Examples of domestic use of ochre in the form of hematite chunks, hematite stained grinding stones and hematite dust on possible house floors were found at the Sheaman site, eastern Wyoming (Frison and Stafford 1982), the Hanson site, Wyoming (Frison and Bradley 1980), the Lindenmeier site, northern Colorado (Wilmsen and Roberts 1978), Stewart’s Cattle Guard site, southern Colorado (Jodry 1987) and the Agate Basin, Wyoming (Frison and Stafford 1982).

Finding Paleo-Indian materials in association with naturally occurring hematite deposits at the Powars II site, Wyoming, Stafford et al. (2003) inferred ochre mining and processing activities. Furthermore, the Folsom age Cooper site, northwestern Oklahoma yielded a bison skull that was decorated with ochre (Bement, L. et al. 1997). Jennings (1989) described the continued use of the mineral through the Archaic in burial contexts, but also the processing of hematite, such as at the Rogers shelter that was used from 10 500 BP to 1000 BP. The Red Ochre complex is another example of ochre use in mortuary context. The Adena peoples used ochre in burial contexts, for example as in the Caldwell’s Little Bluff, southern Ohio (Lovejoy 1975), but also other minerals such as gypsum, a good source of white, which was mined from Salt Cave, Kentucky (Jennings 1989). They stained or coated skeletons with either black or red pigments, using red ochre and graphite.

Furthermore, the Hopewell either heaped pigment onto the dead body or purposefully painted the bones of their dead that were defleshed before internment,
sometimes just hands, skull or feet, and sometimes all of the bones (Webb and Snow 1974: 279). There are many caves in the Eastern Woodlands that have images incised or patterns drawn with different colored materials such as mud. Some date to the terminal Archaic such as the 3rd Unnamed Cave and are associated with mining activity (Simek et al. 1998), while most are associated with the Southern Cult rituals of the Mississippian period including Mudglyph Cave in Tennessee (Faulkner et al. 1984). Another example of colorant use is the tree pictographs (paintings on trees) that occur in different colors and may have functioned as markers.

Iron oxides and hydroxides occurred in a range of colors and crystal structures. Some of these exist as polymorphs, i.e. the same chemical composition has a different crystal structure and a different name. All but magnetite are anisotropic, and impurities such as manganese, clays and organics enhance the variety of colors of natural iron oxide pigments. Due to the great variability of these minerals, the names, elemental composition and colors that they can produce are summarized in table 1. The already mentioned glauconite for green, manganese ore for black and purple, galena and kaolin as a source for gray and white are included, so are several others that have been used historically (Klein 2002).
<table>
<thead>
<tr>
<th>Mineral</th>
<th>Color</th>
<th>Chemical Composition</th>
<th>Possible Traces of</th>
<th>Notes</th>
<th>Occurrence</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hematite</td>
<td>dull to bright red</td>
<td>Fe₂O₃</td>
<td>AL, Ti, Cr, V</td>
<td></td>
<td>Great Lakes, southern Appalachia</td>
</tr>
<tr>
<td>Magnetite</td>
<td>black with metallic luster</td>
<td>Fe₃O₄</td>
<td>Ti, Mg, Ni, Mn, Al, V, Cr</td>
<td>Occurs in context with limestone, hematite, pyrite &amp; other sulfides</td>
<td>New York, Pennsylvania and Arkansas</td>
</tr>
<tr>
<td>Goethite</td>
<td>yellowish to reddish brown</td>
<td>α FeOOH x H₂O</td>
<td>Mn</td>
<td>polymorph of goethite</td>
<td>Great Lakes area</td>
</tr>
<tr>
<td>Lepidocrocite</td>
<td>yellow, red, reddish-brown</td>
<td>γ FeOOH x H₂O</td>
<td></td>
<td></td>
<td>Great Lakes area</td>
</tr>
<tr>
<td>Galena</td>
<td>bluish gray</td>
<td>PbS</td>
<td>Ag, Bi, Sb, Hg, Cu</td>
<td></td>
<td>New York, Missouri, Kansas, Oklahoma, Colorado, and Idaho and Arkansas.</td>
</tr>
<tr>
<td>Kaolin</td>
<td>white, yellow, green, blue</td>
<td>AL₂Si₂O₅(OH)₄</td>
<td></td>
<td>Type of clay</td>
<td>Georgia, South Carolina, North Carolina, Arkansas, New Mexico and New Jersey</td>
</tr>
<tr>
<td>Cinnabar</td>
<td>scarlet or dark red</td>
<td>HgS</td>
<td></td>
<td></td>
<td>Arkansas, Texas, Nevada, California</td>
</tr>
<tr>
<td>Glauconite</td>
<td>yellow-green, green or blue-green</td>
<td>(K, Na, Fe²⁺) 0.33 (Fe³⁺, Al) 1.67 <a href="OH">(Si, Al) 4O10</a> 2</td>
<td></td>
<td>Silicate of iron &amp; potassium, occurs with clay</td>
<td>Dolomite environment of Missouri</td>
</tr>
<tr>
<td>Manganese</td>
<td>black or purple</td>
<td>MnO(OH)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Limonite</td>
<td>yellow, orange, reddish brown or brownish black</td>
<td>Iron oxides</td>
<td>term for mixture of different hydrated iron oxides, mostly Goethite</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Gypsum</td>
<td>white or grey</td>
<td>CaSO₄ · 2 H₂O</td>
<td>Ba, Sr, Mg</td>
<td>Also called hydrated gypsum, lime, gips, selenite</td>
<td>New York, Maryland, Oklahoma</td>
</tr>
</tbody>
</table>

Table 1  Inorganic Mineral Pigments with Elemental Composition
Known Dyestuff and Possible Plant Sources

The introduction provided in the following paragraph related to the colorant chemistry is adapted from *Handbuch der Naturfarbstoffe* by Schweppe (1993). It is supplemented with ethnobotanical data by Moerman (2000, 2005) and phytochemical data by Duke (2005). Additional data concerning plant locals was gathered from the plants database of the United States Department of Agriculture (USDA-NRCS 2005).

Plants, fungi and animal are the sources for organic dyes. Any source (plant or animal) can contain several different chromophores that each produce different colors. On the other hand, a single chromophore can be extracted from several different sources. Therefore, to identify the source of a colorant, several chromophores must first be identified. Then, the determination of the relative ratio of the component chromophores may be necessary to identify the source.

Dyes are often classified into categories according to the chemistry of their attachment to the textile, i.e. direct, vat or metal complex dyes, acid, base, fiber reactive. They are also classified by chromophore, i.e. carotenoid, quinone and others. In the following section, those chromophores that are found in some of North American plants that have been used by native peoples will be briefly delineated.

Carotenoid dyes are considered the most important natural dye category, because they occur in largest quantities among the natural dyes. Carotenoid dyes are fat soluble, nitrogen-rich and they produce all colors between yellow and violet. They are characterized by a string of dehydrated isoprene molecules forming conjugated double bonds, which act as chromophores. The best known compound from this group however is carotene, the colorant from carrots and the precursor of vitamin A, which also gave its
name to this class. Only green plants and some microorganisms (bacteria, fungi, and lower algae) can manufacture carotenoids. According to Schweppe (1993), some of the carotenoid-containing plants that were utilized by the native population containing carotene are pigweed *Amaranthus sp.* (Amaranthaceae), sorrel (*Rumex* sp.), and the little hogweed (*Portulaca oleracea* L.).

The next large dye class that must be considered are the quinones, a family name for dioxo derivatives of dihydroaromatic systems: (1) benzoquinone containing one benzene ring, (2) naphthoquinone containing two benzene rings, and finally (3) anthraquinone containing three (Appendix A, Figure 9).

Schweppe suggests that benzoquinone is a component in about 90 different dyes, which are mostly extracted from plants, but also from fungi. Over 120 dyes can be found in the naphthoquinone group with the best known representative of juglone, which is extracted from the black walnut and butternut (*Juglans* sp.). The anthraquinones are the largest group of the quinones that occur in nature. Many species of the Rubiaceae family contain colorants that belong to this group. The most important dyestuff components of Rubiaceae are alizarin, purpurin and pseudopurpurin. Particular combinations of these anthraquinones distinguish different plant sources (Wouters 1985). Colors produced with anthraquinones differ depending on the dyeing aids that are used. For example, alizarin can be combined with varying strengths of alkali or acids to create different hues. A strong alkali will create a violet-blue color, while a strong acid will create a yellowish red. The color is also influenced by the presence of metallic salts thereby creating a chelate or lake: the alumina lake is rose-red or bluish-red with calcium, the tin lake is red-violet, the iron lake is black-violet, and the chrome lake is brown-violet or red-brown.
Other plants containing anthraquinones are sorrel (Rumex sp.), and other members of the Polygonaceae family.

Most of the yellow colors are produced by the flavonoid dye class, which are all phenols. This group includes flavone, flavonol, isoflavone, flavanone, chalkone and aurone, all containing the same basic chemical structure. The flavonoids contain OH and OCH groups that dictate by their number and position which color will be produced. Flavonoids require a mordant, and these OH and OCH groups form an insoluble lake with the metal ions of the mordant. This generally makes for a waterfast and lightfast color. The plants themselves often contain the flavonoids in the form of glycosides that are separated into a sugar and a colorless or only lightly colored non-sugar (aglycone) by hydrolysis. This aglycone will only develop rich colors after forming a lake with a mordant. Zhang and Laursen (2005) suggest that it is particularly important to account for these and when “analyzing yellow, flavonoid dyes, not only because such dyes are chemically more delicate than red or blue dyes, but also because they can be derived from a greater variety of different plant sources” (Anonymous, 2005). Probably the best known representative from this group is quercetin, which can be extracted from different oak species such as the black oak (Quercus velutina Lam.), but also occurs in onions (Allium sp.) and red grapes (Vitis sp.).

While Schweppe (1993) lists anthocyanine and betalain dyes as a separate group from flavonoids, he also calls them reduced flavonoids. They are sometimes listed as flavonoids, because the basic chemical structure is the same. This colorant group can be used as a pH indicator because it changes from red in acids to blue in bases. The best known plant from which the anthocyanine (sambucin, chysanthemin) constituent can be
extracted is the elder (*Sambucus* sp.). The plant contains many other potential colorants however such as quercetin. Blackberries and dewberries (*Rubus* sp. L), grapes (*Vitis* sp.) and cherries (*Prunus* sp. L.) are some of the other plants that contain constituents of anthocyanine colorants.

Betalain dyes are contained mostly in the flowers and fruits of plants. They received their name from the red dye in beets (*Beta* sp.), but they also occur in other plants such as the pokeweed (*Phytolacca americana* L.). Lambsquarters (*Chenopodium album* L.), a plant that was used extensively by Native Americans in the eastern woodlands, also contains constituents that belong to this dye class.

Basic dyes contain cations of the quaternary ammonia group that will form water-soluble salts with organic or inorganic anions. Berberine (Appendix A, Figure 10) is probably the best known representative of this group. It occurs in many plant species, but for dyeing purposes was mostly extracted from plants belonging to the Berberidaceae family, such as the barberry (*Berberis vulgaris* L.). It also occurs in other plants such as the Oregon grape, golden seal (*Hydrastis canadensis* L.), and bloodroot (*Sanguinaria canadensis* L.). The latter produces other color constituents in addition to berberine.

While the basic dyes contain the quaternary ammonia group, alkaloids contain only secondary or tertiary NH groups according to Schweppe (1993) who lists these separately. Others list both of these as alkaloids.

Historically, tannins and gallotannins were not always categorized as dyes, but rather as tanning agents. A tanning agent is a substance that is able to form non-swelling bonds with the protein of skin transforming it to leather. According to Schweppe, these substances can be very different from each other chemically. Tannins are one of many
types of secondary compounds in plants. They are oligomeric (a polymer consisting of less than five monomers), multiple structure units with free phenol groups. They also have the ability to bind protein and form insoluble and soluble tannin-protein complexes. Tannins are subdivided into two groups: (1) hydrolysable tannins and (2) condensed tannins. Schweppe (1993) calls the first group gallotannins and explains that they are esters of the gallic acid (glycosides). The second group is labeled “condensed tannins”, they do not form esters, but rather they are hydrated flavonols or anthocyanes, sometimes also called proanthocyanidins.

Schweppe (1993) lists ink as an early example of gallotannins combined with green vitriol (hydrated iron sulfate). Gallotannins occur in the woody parts of plants, but also in the galls that grow on leaves, buds and twigs after certain insects lay their eggs onto these parts of the plant. Sumacs (Rhus sp.) are rich in gallotannins and have been used in many parts of the world for tanning purposes. Oak species (Quercus sp.), but also chestnut (Castanea sp.) and alder trees (Alnus sp.) are good sources for gallotannins.

The condensed-type tannins also occur in the woody parts and bark of plants that belong to the Pineaceae family such as pine, spruce, larch, hemlock, but also oak and willows and others.

Indigoid dyes are often mentioned as source for blue. A variety of plants have been used to produce indigoid dye. Most natural indigo is obtained from plants in the genus Indigofera. While it must be remembered that the “indigo” plant (Indigofera tinctoria L.) is an introduced species, some native plants of that genus exist, such as coastal indigo (Indigofera miniata Ortega), Carolina indigo (Indigofera caroliniana P. Mill), or anil de pasto (Indigofera suffruticosa P. Mill).
Background for the Chosen Methods

In the following section, the background theories of the methods that were employed are briefly summarized. It is important to consider that the least invasive methods such as visual examination and photography are used first, while the more destructive methods are only used when other possibilities are exhausted.

Photography

The wavelengths of the electromagnetic spectrum (Appendix A, Figure 11) from 400-700 nm are visible to the human eye. They range from the blue light at 400 nm through red at 700 nm. The ultraviolet (UV) region is comprised of wavelengths less than 400 nm and extends into the x-ray region, while the infrared spectrum is composed of frequencies of more than 700 nm. Both of these spectra are not visible to the human eye, but their use in photography enables the observer to see in these otherwise non-visible regions and are used extensively as tools to recover more information about the subject.

There are two types of ultraviolet photography: reflected or direct, and UV fluorescence. In both, the sample is exposed to UV light.

UV Reflected Photography

A light source emitting ultraviolet light is directed at the subject which will then reflect this radiation into the camera. With the reflected technique, it is necessary to fit an ultraviolet transmission filter over the camera lens to prevent any visible radiation from reaching the film (Appendix A, Figure 12) Visible radiation from the room or reflected
from the subject will be absorbed by the filter over the camera lens, and hence can be ignored.

**UV Fluorescence Photography**

The second type of UV photography involves the discharge of electron excitation or the fluorescence of a substance when excited by UV illumination. The absorption of UV light causes the electrons of the subject's atoms to be temporarily raised to higher energy orbits. The electrons then naturally slip back to their normal orbit releasing energy in the form of light, which always has a wavelength of lower energy than the excitation source, in this case the UV light. Thus, subjects irradiated with ultraviolet may release, for example, green, yellow or pink light and subjects irradiated with visible light may emit infrared fluorescence.

To capture this fluorescence, the source of ultraviolet radiation filtered with an ultraviolet transmission filter - or excitation filter - is aimed at the subject in a completely darkened room (Appendix A, Figure 13). The subject reflects the ultraviolet light, but can also emit a visible fluorescence. The ultraviolet light is then prevented from reaching the film by a barrier filter that will only allow visible light to be transmitted. This technique is used in forensics, ophthalmology, and oncology among others. Forensic scientists use UV light to detect forgeries, but it is also used in the textile industry to detect manufacturing defects. Furthermore, materials such as pigments, iron stains and paint found on a textile may fluoresce. The presence of certain dyes can be determined based on their fluorescence (Andrew and Eastop 1994). For instance, “one of the oldest organic pigments to be found is the natural madder lake…of alizarin…but also containing purpurin (1,2,4-trihydroxyanthraquinone), [which] fluoresces in a bright yellow-
red...[which is] activated by energy of 400 nm...[and] weaker fluorescence at 345 nm (Johnston-Feller 2001:207). Berberine is another example of a natural dye that fluoresces (Hayashi 1978:48).

\textit{Infrared Photography}

In infrared photography, the subject is exposed to light that contains the infrared spectrum such as a tungsten light or photoflood lamp. The visible part of the spectrum is filtered out by using an IR transmission filter, which transmits a specific energy range from the IR region (Appendix A, Figure 14). An IR sensitive film has to be used to record images.

In conservation, paintings are often examined with IR illumination because pigments and varnishes vary in their reflectance and absorption in that portion of the electromagnetic spectrum. The wavelength is longer than visible light; therefore, the small particles in pigments and varnishes scatter the light less efficiently than visible light. IR can be used to “reveal underpaint, underdrawing in carbon black, retouching, and changes in composition as well as distinguish and penetrate beneath discolouration or soiling”. The different reflectances of vegetable dyes can be used when examining textiles for evidence of restoration for instance (Andrew and Eastop 1994).

\textbf{Use of Microscopy for Archaeological Textile Materials}

Techniques of optical microscopy are well known and often used for the examination of fibers and yarns from archaeological textiles. Many texts provide the fundamentals of the technique (e.g. McCrone 1984; McCrone, Slayter and Slayter 1992). Additional manuscripts in the literature describe the methods that may be employed in
the study of forensic materials (Petraco 2004) and archaeological materials in particular (Jakes 2000; Curl and Jakes 2003) while many others report the results of optical microscopic examination in identification and characterization of fibers (Gremillion, Jakes and Wimberley 2000; Song, Jakes and Yerkes 1996).

Similarly, scanning electron microscopy is a well known method of examining and imaging fibers and particles. Texts such as Goldstein et al. (1992) provide the fundamentals of the technique. Many manuscripts in the literature report the use of this technique in the study of archaeological materials (e.g. Chen, Jakes and Foreman 1996; Sibley and Jakes 1996).

Optical Microscopy (OM) and Scanning Electron Microscopy (SEM) complement each other in the ability to capture images at different magnification, depth of field and resolution. In OM, magnification is limited by the nature of the light and the numerical aperture of the objective that is used. It can however be used to study both surface and interior structures. The interaction with the light often provides additional information, including relative crystallinity, and refractive indices of the examined materials. Fibers may be identified by morphology, optical behavior and micro-chemical reactivity. In SEM, only the surfaces of the specimen may be observed but the depth of field is much greater than in OM allowing a magnification of up to ~ 100 000X. Surface features such as residual marks left when scales on hair fibers have degraded, can be detected. Typical SEMs require a high vacuum, and carbon, gold or platinum coating of the samples to increase conductivity and a special detector. New environmental SEMs allow examination under atmospheric pressure and moisture conditions.
SEM is particularly useful when integrated with an energy dispersive spectrometer (EDS), thereby allowing the determination of elemental composition of the materials that are also being observed and micrographed. Elemental composition of fibers and deposits have been studied in textiles from Etowah (Sibley, Jakes and Song 1989). The elemental composition reflects their burial environment in association with copper as well as their constituent plant fibers. Rowe (2001) applied this technique successfully to pigments used in rock art, and it has been used in the study of archaeological fibers (Song 1991; Jakes and Mitchell 1996; Srinivasan and Jakes 1997; Chen and Jakes 2001).

Detection of Inorganic Constituents

Inorganic pigments and lakes (organic dyes bonded to an inorganic support) can be recognized by the ratio of elements in their composition, making elemental analysis an important tool in their identification. EDS may facilitate an initial qualitative analysis, but quantitative analysis and the detection of trace elements is needed to identify the inorganic colorant components. Due to sample size restrictions, the methods that can be employed are limited. However, some of these quantitative methods such as spectrometry can be used. Spectroscopy is the study of spectra with many different delivery systems to prepare the sample and detection systems to acquire the spectra exist. Herein, only mass spectrometry (MS) which measures characteristic mass and optical emission spectrometry (OES) which measures light are described. Spectrometry comprises several techniques that form an important means for the chemical analysis of inorganic elements.

The inductively-coupled plasma (ICP), which prepares the sample to be analyzed, is a very powerful ion source. Because the source operates at temperatures of 7000 K,
almost all molecules in a sample are broken up into their component atoms. In ICP, a radio frequency (RF) signal is fed into a tightly wound, water-cooled coil where it generates an intense magnetic field. In the center of this coil is a specially made glass or quartz plasma torch where the plasma is formed. The plasma, which is defined as a gas consisting of ions, electrons, and neutral particles is generated in an argon gas. The behavior of the gas is dominated by the electromagnetic interaction between the charged particles. Thereby, the plasma atomizes and ionizes the elements in a sample, and the resulting ions are then passed through a series of apertures (cones) into an analyzer (MURR 2005).

In optical emission spectrometry (OES), emitted spectra by atoms and ions with optical transitions in the wavelength range from about 100 nm to 900 nm are measured. This range includes the ultraviolet, and visible light (from violet at 380 nm to red at 760 nm), and the near infra-red. Such emissions are characteristically different from emissions at very short wavelengths (x-rays and gamma rays), and from emissions at long wavelengths (far-IR and radio waves). In particular, different types of instruments are used to detect them. Optical emissions are generally detected with optical spectrometers.

Mass spectrometers (MS) use the difference in mass-to-charge ratio (m/e) of ionized materials to separate atoms or molecules from each other. The intensity of a specific peak in the mass spectrum is proportional to the amount of that isotope (element) in the original sample. There are different detectors in use: quadrupole and high resolution. They differ in their resolution. Both systems use a combination of pulse counting electron multiplier and analog detectors to ensure a wide linear dynamic detection range from parts per quadrillion (for some elements) up to hundreds of parts per
million. The high resolution detector is also called double focusing detector because of its ability to focus the ions by energy and mass/charge ratio. These systems use a powerful electro-magnet with a varying magnetic field instead of an RF/DC field as used in a Quadrupole (MURR 2005).

Before introducing a sample to any of these instruments, it must be digested in a solvent, which is typically acid or a combination of acids. The complete digestion of the sample material is very important for the effective operation of the instrument, and differs depending on the sample and the expected content.

ICP spectroscopy has been applied to do quantitative elemental analysis in forensic examinations (Robertson 1999), and for such issues as the determination of source provenance based on these data for materials such as ochre (Weinstein-Evron, M. and Shimon Ilani 1994) or other pigments.

Besides digesting samples, laser ablation is another way of introducing the sampled material to the ICP-MS. In LA-ICP-MS, a laser pulse vaporizes and ionizes at the same time on a very small area on the sample. This brings an additional advantage of selective sampling, because it creates a point specific micro-sample that is subject to the analysis (MURR 2005). Speakman et al. (2002) reports on the characterization of archaeological materials with this technique, while others analyzed pigments successfully on pottery from the American Southwest (Sall et al. 2005), Mesa Verde region (Speakman and Neff 2002), Mexico (Rodriguez 2002), and Peru (Vaughn et al 2002, 2005).
Detection for Organic Constituents

There is a vast body of literature focused on the identification of dyes. Verhecken (2005) provides a good review of the types of methods used in the past for colorant identification. He classifies these as: 1) chemical methods which might yield colorimetric results, 2) chromatography, which separates components of differing chemical composition, and which requires some subsequent method of detection of the separated chromophores, and 3) spectroscopy, including visible, UV-visible, fluorescence, infrared, Raman, mass, and nuclear magnetic resonance techniques. No single technique is outstanding in its performance, each has advantages and disadvantages. In fact, Wouters (2005) states the need for further development of colorant identification procedures that address issues raised by currently used methods. Since the concentration of dye on a fiber is relatively small, colorant identification requires the identification of a very small quantity on a material whose sample size is very limited. In addition, some methods employed require dissolution of the material, which breaks dye-mordant complexes and thus might cause some alteration that would affect identification. Finally, dyes can be complex mixtures of multiple colorants, thus the determination of relative ratios of these components is needed in order to distinguish their sources. Most research has focused on the identification of typical colorants used in Europe and the Middle East, with some work on those used in the Far East, and in South America. Little work has been done with North American colorants. Reliable plant and animal sources that will serve as comparative materials are needed for accurate determination of the unknown colorants of North America. Very little is known concerning North American colorants and their use and much more needs to be done in the field of colorant identification. Therefore, the
protocol developed herein does not indicate one single particular analytical method for
the identification of organic colorants. The protocol provides guidance for the sample
taking once the methods can be developed and tested.
CHAPTER 3

METHODS USED TO DEVELOP THE IDEAL PROTOCOL

Development of the Protocol

In this chapter, the methods of examination used in this research are described. To confirm the efficacy and suitability of the chosen methods, two sets of simulations of coloration anticipated on prehistoric textiles were produced in the lab. These were used to test all methods and to develop the actual protocol. Finally, the ideal protocol is presented.

Replicas for the Pre-Tests

Either painting or dyeing would have been used to apply color in prehistory. For the pretest, two sets of simulations were created, painted fabric and dyed fibers. Both materials were then used to pretest the equipment for UV fluorescence and UV reflected photography and to evaluate the fluorescence of the materials that had been applied on these substrates. These materials were also used as comparative materials for the microscopic examination at a later time in the research.

Painted Fabric

To carry out this simulation, three components were needed: a fabric (substrate), a binder and a colorant that would fluoresce when exposed to a UV light source. Linen
(Testfabrics L-57). [Note: Full names and addresses of suppliers are listed in Appendix G] was chosen as a substrate for the simulations because flax is a bast fiber; comparable to the fibers used in prehistory, such as milkweed and Indian hemp. Furthermore, the main component of flax is cellulose as is that of milkweed or Indian hemp. Small bands of different potential colorant sources, both mineral based and organic, were painted on the linen testfabric. The colorants used were ferrous oxide, an alizarin/purpurin mixture, ground up bloodroot (*Sanguinaria canadensis* L.) and bedstraw (*Galium* sp.). As carriers or binder for the pigment colorants to be painted on the fibrous materials beef fat was used as a substitute for bear grease and egg white was used as source for albumin. The various combinations of these materials were mixed and painted on areas of the testfabric as outlined in table 2 (Appendix C, Table 2). The painted testfabric (Appendix A, Figure 15) was left to dry for 24 hours.

**Colored Fibers**

Ochre is mentioned extensively in the archaeological literature as having been used for different applications in prehistory, coloration being one of them. Therefore, rabbit hair and milkweed fibers were both colored with an aqueous solution of lab grade hematite (Fe$_2$O$_3$) as a substitute for ochre (see glossary). Additionally, rabbit hair from a breeder (Jennings, T.), commercially produced rabbit yarn (Joseph Galler Inc.), and milkweed fibers that had been collected by the researcher in the fall of 2004 were also colored in an aqueous bloodroot dye bath that did not contain any dyeing aids. Since berberine fluoresces in the UV region, it constituted an ideal choice for checking fluorescence in a dyed material. Copper sulfate in an aqueous solution was used to color
milkweed and rabbit hair. It was checked for fluorescence, but it was also later used in microscopy as comparative material.

<table>
<thead>
<tr>
<th>#</th>
<th>Colorant</th>
<th>Binder</th>
<th>Aid</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Fe$_2$O$_3$</td>
<td>Beef Fat</td>
<td>None</td>
</tr>
<tr>
<td>2</td>
<td>Fe$_2$O$_3$</td>
<td>Albumin</td>
<td>None</td>
</tr>
<tr>
<td>3</td>
<td>Alizarin/purpurin</td>
<td>Beef Fat</td>
<td>None</td>
</tr>
<tr>
<td>4</td>
<td>Alizarin/purpurin</td>
<td>Albumin</td>
<td>None</td>
</tr>
<tr>
<td>5</td>
<td>Alizarin/purpurin</td>
<td>Beef Fat</td>
<td>Tannic Acid</td>
</tr>
<tr>
<td>6</td>
<td>Alizarin/purpurin</td>
<td>Albumin</td>
<td>Tannic Acid</td>
</tr>
<tr>
<td>7</td>
<td>Dried, ground bloodroot</td>
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<td>None</td>
</tr>
<tr>
<td>8</td>
<td>Dried, ground bloodroot</td>
<td>Albumin</td>
<td>None</td>
</tr>
<tr>
<td>9</td>
<td>Dried, ground bloodroot</td>
<td>Beef Fat</td>
<td>None</td>
</tr>
<tr>
<td>10</td>
<td>Bedstraw</td>
<td>Beef Fat</td>
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</tr>
<tr>
<td>11</td>
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<td>Albumin</td>
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</tr>
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<td>12</td>
<td>Albumin only</td>
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</tr>
<tr>
<td>13</td>
<td>Bloodroot dyed milkweed fibers</td>
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<td>None</td>
</tr>
<tr>
<td>14</td>
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<td>Undyed Rabbit hair yarn</td>
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<tr>
<td>16</td>
<td>Fe$_2$O$_3$ colored milkweed fibers</td>
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<td>18</td>
<td>Bloodroot dyed rabbit hair</td>
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<tr>
<td>21</td>
<td>Copper sulfate colored milkweed</td>
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</tr>
</tbody>
</table>

Table 2 Substrate and Colorant Combinations used as Comparative Materials

Photography

To determine the best photographic method for the study of prehistoric textiles, experimentation with different light sources was necessary.

A single lens reflex (SLR) film camera (Canon AE1) with an option for manual adjustments, and different macro and zoom lenses and filters were used for all photography. The camera was mounted on a copy stand with the lens parallel to the textiles to facilitate longer exposure times without losing sharpness of the acquired
images. All pictures were taken with the samples placed on flocking paper (Edmund Scientific). This creates an optically inert background and does not reflect any light, and therefore does not interfere with any optical properties that the textile to be examined might have.

In addition to the effectiveness of a UV light (SIRCHIE, Inc.) as an excitation source, portability and expense of the light sources were factors considered in the acquisition of the equipment. Several light sources were purchased to simulate different lighting conditions: Xenon flash, which emits a wide range of electromagnetic radiation, mainly mid-infrared to shortwave UV, to simulate daylight conditions (6420 K), and a photoflood or reflector flood lamp (3400 K), which is a good source of infra-red light, were evaluated. Moreover, a forensic long-wave ultra-violet light source (~10 000 K) was acquired. The textiles were then photographed in these different lighting conditions: visible light, ultra-violet (UV) light (365 nm) and infra-red (IR) light (>700 nm).

Great care must be taken to limit exposure time of artifacts to excessive UV and IR radiation so as not to accelerate degradation unnecessarily. Furthermore, protective gear (goggles or UV face shield, gloves and long-sleeved clothing or lab coat) was worn at all times to avoid personal injuries due to exposure to the UV light. Also, frequent breaks had to be taken during the shooting sessions using the UV, because it produces ozone, and to keep the overheating of the light source at a minimum.

*Ultra-Violet (UV) Fluorescence and Reflectance*

For the UV lighting conditions, a pre-test using the replicated materials was conducted to determine which lamp would work best as an excitation source to stimulate fluorescence, and to narrow down the exposure times, film speed, and aperture size
before dealing with the actual artifacts. The room was darkened. Only the manual SLR camera (Cannon AE1) was used and Kodak Gold 100 film for colored prints was the medium. Fluorescence generally occurs in a range of the visible spectrum so that different UV cutting filters can be used. Otherwise, the photographs turn out to be very blue and no longer represent the actual fluorescing colors. For this research, the Kodak Wratten # 2E filter was used, because the fluorescing colors occurred mostly in a bluish-white. If the fluorescence appears in different colors, the following filters (B&H Photo) are good options to capture images the reflect these colors properly: Kodak Wratten # 8 for yellow, and a light balancing filter Kodak Wratten # 81 EF for green, yellow and red fluorescence.

For the UV reflectance photography, a UV transmission filter was acquired, i.e. the visible light range is blocked, while the UV light is passed through the filter. Since the Kodak Wratten #18, which is most frequently mentioned in the scientific photography literature, is no longer made, a B+W 403 [Schott UG 1] (The Filter Connection) was obtained. This filter is very similar in transmission range to the Kodak filter. Although this dark-violet filter transmits a small amount of visible light (400-410 nm), it completely blocks wavelengths longer than 410nm.

As for any close-up photography, a medium to small f-stop was used, and the exposure time was bracketed to maximize results.

**Infra-Red (IR)**

To sort out the conditions necessary for IR photography, the replicated materials were used to pretest the methods in the laboratory as was done for the UV conditions.
The photoflood light (B&W Photo) was used as the light source, because it is rich in red light (3400 K) and emits some IR light as well. When used with regular film, the pictures show warmer colors. IR conditions only occur and are captured when the proper film and the proper filter are used.

The pictures were taken with high speed IR black and white film (B&W Photo) by Kodak (HIE), which is sensitive to light at about 900 nm. The film must be put into the camera in total darkness to avoid accidental exposure. The Cannon AE1 was used. The Canon Rebel 2000 was not suitable for this application due to its internal LED components, which fogs up IR sensitive film. A Kodak Wratten 87C filter (B&H Photo) was required for the camera, because IR sensitive film is also sensitive to violet, blue, and red light. The 87C effectively cuts all light frequencies below 800 nm, and thereby facilitates only IR with a range from 800-900 nm. Other filters that are used for IR photography still transmit some red light (# 25 > 580nm; #29 > 600nm; # 70 > 660 nm; # 89B > 700 nm; # 88A, # 87 > 750 nm), which was not desirable for this application.

Extensive bracketing is necessary, because light metering through the filter is not possible. Therefore, the camera must be focused before the filter is put in front of the lens. A lens holder (The Filter Connection) that allows for the filter to be moved in front of the lens and removed again without having to be manipulated otherwise is most practical for this application, so the image will remain in focus. The room does not need to be darkened since all light below 810 nm is cut by the filter. Putting the filter in front of the lens changes (1) the focal point and (2) the effective film speed for which adjustments have to be made on the camera:
(1) To adjust the focal point to IR conditions, the camera must be adjusted after it has been focused. In most cameras, this adjustment is marked on the objective with a red dot.

(2) Using the # 87C filter slows the speed of the film down. Therefore, this change in film speed must be considered when bracketing. In the case of the Kodak film that was used for this research, the film speed slowed from ISO 400 to an effective film speed of about ISO 10. This differs from film to film, and can be looked up on a chart that comes with the film and must be considered when bracketing for aperture size and shutter speed. To determine bracketing settings, a good starting point is set by the formula: f-stop 16 at 1/effective film speed. For example: f-stop16 at 1/8, 1/15, 1/30, 1/60; f-stop 11 at 1/15, ..., 1/125; f-stop 8 at 1/30, ..., 1/250; f-stop 5.6 at 1/60. . . .

**Proposed Sample Sectioning**

The condition of the material to be examined dictates the manner in which the material must be sampled and subdivided for further analysis (Appendix A, Figure 16). In the ideal situation, small samples are subdivided for individual tests to maximize the use of each sample. These sub-samples should provide enough material to perform different analytical examinations, beginning with microscopic study and prioritizing inorganic or organic analyses. Possibilities for inorganic analyses of small samples include several spectroscopy methods (EDS, ICP-OES, ICP-MS, LA-ICP-MS). Organic analysis of small samples could entail different types of chromatography tied to a detection system such as gas chromatography mass spectrometry (GC-MS), while molecular spectroscopy such as
Micro Infrared (IR) and Micro Raman techniques could be used to identify chemical species, determine crystallinity and facilitate profiling of materials of both organic and inorganic composition. Complimentary data can be gleaned from the appropriate sequence of sample selection, subdivision and analyses.

If the textiles are severely degraded and/or mineralized, and the specimen is severely brittle and fragile, it may not be possible to subsection already acquired samples. Instead, it may be necessary to take several very small samples, possibly only fibers, directly from the textiles. Methods are those that are sensitive to concentrations of materials in small samples.

Analysis

The following section describes the results in the protocol testing, particularly in areas of optical microscopy (OM), scanning electron microscopy (SEM) and the elemental analyses.

Optical Microscopy and Imaging

For OM analysis, a Zeiss Axioplan Microscope, with polarized light and differential interference capabilities was utilized. Brightfield (BF), darkfield (DF), polarized light (P) and differential interference contrast (DIC) techniques were used to aid visualizing the morphology of the fibers and the particulates on the fiber surfaces. A 10x eyepiece and 10x and 40x objectives were used. For the image capture and analysis a ProgRes 3008 Digital Camera, Adobe Photoshop version 5.0 and Zeiss AxioVision version 3.1 software were used.
**Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS)**

A JEOL JSM-820 with an Oxford eXL energy dispersive X-ray analyzer were employed in examining the fibers at higher magnifications than the light microscope. SEM–EDS were conducted with the assistance of Dr. Sreenivas Bhattiprolu from the Microscopic and Chemical Research Center located at the Department of Geological Sciences at the Ohio State University. The samples were collected and put onto an aluminum planchette covered with double sided carbon tape (SPI). These were then introduced into the chamber of a Denton Vacuum Desk II coater to be carbon coated. While images were collected, the objective was not to identify the fibers themselves beyond their classes of being hair or bast fiber. Rather, the aim was to capture images and to magnify the fibers enough that adhering deposits could be separated from the fibers and an EDS analysis could be performed on the deposits alone and fibers alone. EDS allows for the semi-quantitative measurements of available elements. The carbon peaks were compared to the Zero peak, which shows if organics are present in the sample in addition to inorganic elements. To collect better images at higher magnification, the samples would be gold or platinum coated.

**Elemental Analysis by Mass Spectrometry**

This elemental analysis was performed using the Perkin-Elmer Sciex ELAN 6000 Inductively Coupled Plasma—Mass Spectrometer (ICP-MS). The samples must be digested to bring them into solution, and controls must be available, hence the EDS must be performed on materials of unknown composition before the ICP-MS. Nitric acid (HNO₃) was used to digest the samples of the simulations colored with ferrous oxide.
The iron oxide colored fibers (Table 2, #16, 17) were weighed into the following aliquots: (1) 1 mg rabbit hair (RH), (2) 5 mg RH, (3) 1 mg of milkweed (MW), (4) 5 mg MW and (5) a mixture of 2.5 mg RH and MW each. These samples were combined with concentrated HNO₃, and the mixture was warmed up to just below boiling temperature for 5 to 7 minutes. Then these samples were analyzed in the spectrometer.

To keep the sample size as small as possible and to measure Fe, the ICP-MS was evaluated before that of an ICP-OES, which requires higher concentrations or a much larger sample size. However, the quadrupole MS also did not have the resolution to measure the main isotope of iron (Fe 56) in these small concentrations. Therefore, the use of the higher resolution spectrometry instrument was found to be necessary if the identification or iron containing minerals is desired (Anthony Lutton, personal communication, 5-9-2005).

Results of the Protocol Development: The Ideal Protocol

This section describes the results of the pretests employed to evaluate the suitability of methods for their ultimate application to prehistoric textiles and fibers. The methods that worked well in addition to the sequence of these methods constitute the ideal protocol.

Photography

To pretest the effectiveness of the light sources and lab conditions necessary for the photographic components of the research, each prepared replica was exposed to different light sources in the Infra-red (IR) and Ultraviolet (UV) range.
UV Light Sources

Initially, a dual frequency UV light source was used with a 4 Watt output (782ADC, Forensic Lights-Dual Wavelength UV Lights). This light caused excitation of the colorant materials on the replicas and therefore resulted in fluorescence. However, the distance needed between the light source and the test materials had to be very close to generate fluorescence. This need for a very short distance (3 to 5 cm) made this lamp unsuitable as a source for fluorescence photography. However, it may be a good choice if photography is not part of the examination, because it is small, portable, relatively inexpensive, and yet it covered the long range as well as the short range within the UV spectrum.

The second light source that was tried was also a dual UV light with a 6 Watt and 12 Watt output for short wave (254 nm) and long wave (365 nm). These generated enough fluorescence to recognize it easily, but the distance between the sample and the light source was still not large enough to take pictures.

As another UV source, two separate lamps (SW969 Trimester Imager-Lighting (254 nm), again with the short and the long wave lengths (UV9691 Companion Illuminator, 365 nm) were tried. This time the output was 6 Watts and 12 Watts. It was recognizable that the 12 Watt output light worked better than the 6 Watt light. However, the distance between the light source and test materials needed to be very close (15-20 cm) to generate fluorescence. It was also determined that 254 nm did not generate fluorescence on the prepared sample. However, fluorescence of dust particles and other contaminants was achieved, which confirmed that the light source worked correctly.
Finally, an even more powerful light source was used (TIGERUV™ Light, 110V AC, 365 nm). The output is 100 Watts and fluorescence was achieved easily. The distance between substrate and light source still needs to be relatively short (30 to 40 cm) but it is suitable to accommodate photography.

As a more portable device, a UV flashlight (INOVA X5MT-UV) with five LED that are surrounded by reflectors was acquired. The flashlight stimulates fluorescence at 395 nm, but is unsuitable as the only light source for photography. However, it is very portable and allows for a quick fluorescence check of artifacts without having to set up the other light on a tripod as required by the larger lamps such as the TIGERUV™ Light.

**UV Fluorescence**

Milkweed and rabbit hair fibers that had been dyed with bloodroot were examined under the UV light source to determine if fluorescence was produced. The milkweed fibers dyed in bloodroot fluoresced in a pale yellow/orange. The rabbit hair yarn fluoresced in a bright yellow/orange as well. However, the undyed rabbit hair yarn also fluoresced intensely. It was concluded that the commercially acquired rabbit hair yarn was more than likely treated with a fluorescent optical brightening agent, which is commonly done in industry.
<table>
<thead>
<tr>
<th>#</th>
<th>Colorant</th>
<th>Binder</th>
<th>Aid</th>
<th>Fluorescing</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Fe₂O₃</td>
<td>Beef Fat</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>2</td>
<td>Fe₂O₃</td>
<td>Albumin</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>3</td>
<td>Alizarin/purpurin</td>
<td>Beef Fat</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>4</td>
<td>Alizarin/purpurin</td>
<td>Albumin</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>5</td>
<td>Alizarin/purpurin</td>
<td>Beef Fat</td>
<td>Tannic Acid</td>
<td>None</td>
</tr>
<tr>
<td>6</td>
<td>Alizarin/purpurin</td>
<td>Albumin</td>
<td>Tannic Acid</td>
<td>None</td>
</tr>
<tr>
<td>7</td>
<td>Dried, ground bloodroot</td>
<td>None</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>8</td>
<td>Dried, ground bloodroot</td>
<td>Albumin</td>
<td>None</td>
<td>Pale yellow</td>
</tr>
<tr>
<td>9</td>
<td>Dried, ground bloodroot</td>
<td>Beef Fat</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>10</td>
<td>Bedstraw</td>
<td>Beef Fat</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>11</td>
<td>Bedstraw</td>
<td>Albumin</td>
<td>None</td>
<td>Orange or pale salmon</td>
</tr>
<tr>
<td>12</td>
<td>Albumin only</td>
<td>None</td>
<td>None</td>
<td>Bluish white</td>
</tr>
<tr>
<td>13</td>
<td>Bloodroot dyed milkweed fibers</td>
<td>None</td>
<td>None</td>
<td>Pale yellow</td>
</tr>
<tr>
<td>14</td>
<td>Bloodroot dyed rabbit hair yarn</td>
<td>None</td>
<td>None</td>
<td>Bright pale yellow</td>
</tr>
<tr>
<td>15</td>
<td>Undyed rabbit hair yarn</td>
<td>None</td>
<td>None</td>
<td>Bright bluish-white</td>
</tr>
<tr>
<td>16</td>
<td>Fe₂O₃ dyed milkweed fibers</td>
<td>None</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>17</td>
<td>Fe₂O₃ dyed rabbit hair</td>
<td>None</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>18</td>
<td>Bloodroot dyed rabbit hair</td>
<td>None</td>
<td>None</td>
<td>Pale yellow, weak</td>
</tr>
<tr>
<td>19</td>
<td>Undyed rabbit hair</td>
<td>None</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>20</td>
<td>Copper sulfate colored rabbit hair</td>
<td>None</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>21</td>
<td>Copper sulfate colored milkweed</td>
<td>None</td>
<td>None</td>
<td>None</td>
</tr>
</tbody>
</table>

Table 3 Substrate and Colorant Combinations—Results of Fluorescence Test

From these results, it was concluded that the rabbit hair yarn was not suitable as control for fluorescence photography, because the undyed rabbit hair fluoresced by itself. Hence, untreated rabbit hair was obtained directly from a breeder to continue the pretest and to use as control.

After the lighting conditions were established, pictures were taken using ISA 100 Kodak Gold film, and a Wratten 2E filter, which blocks frequencies below 405 nm. The
colors in the pictures without this filter are very blue, which distorts the fluorescing colors severely. A Canon AE1 camera with macro (nominal 2x) lenses was used, and the distance between camera housing and sample was 35 cm. After extensive bracketing, it was established that the best results were achieved at a f-stop of 5.6 with exposure time between 4 and 8 sec. The same photography was repeated using a Canon Rebel 2000. The results were the same, but the macro lenses were not necessary after the distance between sample and camera was changed to 50 cm and the zoom lens on the camera was used. Both SLR cameras are good choices for this application.

*UV Reflectance*

The basic set-up was similar to the fluorescence photography set-up in that the camera was mounted at a distance of 50 cm, and Kodak Gold Print ISA 100 film was used. The camera needed to be focused before the filter was attached, since all visible light was blocked out. Extensive bracketing established that the best results were achieved at a f-stop 5.6 and exposure time between 5 and 8 minutes.

*Infra-Red*

The camera was mounted at a distance of 50 cm, and a Kodak HIE high speed black and white infrared film was used. The camera also needed to be focused before the filter was attached. Extensive bracketing with these replicated textiles established that the best results would be achieved at a f-stop 5.6 at 1/250 and 1/125 sec exposure. However, to gain maximum depth of field, different aperture sizes still needed to be used. Therefore, the artifacts still need to be photographed according to the following
specifications: f-stop 8 at 1/125 and 1/60 and at f-stop 16 at 1/30 and 1/15 sec exposure time in addition to an f-stop 5.6 at 1/250 and 1/125 sec.

**SEM Imaging, EDS and ICP-MS/ICP-OES for Elemental Analysis**

SEM-EDS was not performed with the replicated samples before using it on the actual artifacts, because it has been applied frequently to analyze historic and archaeological fibers and to do a qualitative elemental analyses.

However, in the study of archaeological fibers it is crucial that EDS be performed **before** moving on to ICP-MS or ICP-OES. Furthermore, the objective of the protocol was not only to get a quantitative elemental analysis, but also to keep the sample size as small as possible. Therefore, the EDS must be considered a preliminary test for the elemental analysis by ICP-MS or OES, thus allowing for the chemical composition of the material to dictate what must be done next.

Concentrated Nitric acid (HNO₃) was used to digest the samples of fibers previously colored with ferrous oxide. This mixture was warmed up to just below boiling temperature (70° to 75 °C) for 5-7 minutes, which did not result in total digestion. Hence, the incubation time in the water bath was increased to 30 minutes.

The samples were analyzed using the Perkin-Elmer Sciex ELAN 6000 Inductively Coupled Plasma Mass Spectrometer (ICP-MS). However, this instrument’s resolution was not high enough to measure the quantities that needed to be analyzed for the iron ions that were supposed to be measured. Therefore, the ThermoFinnigan Element 2 Inductively Coupled Plasma Sector Field Mass Spectrometer was used to do further analysis. While using the Element 2 instrument, the sample dilute clogged up the tubing to the instrument several times. It was concluded that the samples were only digested
enough that a visual examination could no longer see fibers in the dilute. However, they were not digested enough to pass through the tubing, and therefore, the digestion must be adapted, while keeping such issues as matrix effect of the chosen acid in mind.

The results of this section and the sequence in which the steps of the protocol must be applied are summarized in Figure 17 (The Ideal Protocol and Sequencing Flowchart, Appendix A). Through extensive visual examination and forensic photography of archaeological textiles, regions of particular interest may be determined, even when these are not visible to the naked eye. Informed by these photographs, samples may be selected which are more likely to contain important information about the chemistry of the fabric. That is, if there are variations in the chemistry due to coloration for example, forensic photography aids in displaying that variation and representative samples can be chosen. Sequencing of subsequent examinations and analyses maximizes the data obtained from a single small sample.

Once a sample is taken, further study under low magnification macroscopy can reveal additional features including color or structure variation. This information aids in the selection of sub-samples. Through OM of fibers and particulate, identifications can be made. Furthermore, the general type of colorant can be determined by its location, i.e. pigment on fiber surfaces or dyes with color thoroughly impregnating the fibers. A single small fiber which has been chosen in an informed manner, can yield not only morphological information through microscopic study, but also elemental composition through EDS. The resulting EDS data of the fibers and of particulate adhering to the fibers provide information about the ratio of inorganic elements. These in turn lead to determination of subsequent analyses for organic or inorganic components, because
informed choices are possible. For example, the solvents needed to digest sampled material and the controls for ICP-MS can be determined without wasting material. On the other hand, a high carbon content would require methods detecting organic components such as chromatography or molecular spectroscopy, micro IR or Raman.
CHAPTER 4

METHODS USED FOR THE SEIP TEXTILES: APPLICATION OF THE PROTOCOL

This chapter describes the results of the application of the protocol to the actual Seip textiles. The visual examination of textiles, selection of representative textiles for in-depth study, acquisition of samples, and the sequence of examination are also described.

Selection of Samples Suited for Analyses

Permission to take photographs and remove samples of the selected Hopewell textile fragments was requested from the appropriate entities at the Ohio Historical Society (OHS). There are a total of 226 Seip textiles curated at the Ohio Historical Society. Only those textiles that did not directly adhere to copper or were otherwise not directly associated with copper were examined for evidence of coloration. These were divided into three groups based on patterns in: (1) yellow/brown, (2) white/turquoise, and (3) black on black patterns on charred textiles that may indicate previous patterns of coloration. Eleven textiles representing each of these groups (four yellow/brown, two white/turquoise and four charred) were selected for photography and sampling. Hereinafter this group will be labeled the “selected” textiles. In addition, one textile that upon visual examination did not show any apparent coloration was chosen as a “non-colored control” (Table 4).
<table>
<thead>
<tr>
<th>Textile # (Song)</th>
<th>Textile # (Thompson)</th>
<th>Apparent Color</th>
<th>Charred Yes/no</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>0701'-'231</td>
<td>Yellow/Brown</td>
<td>No</td>
</tr>
<tr>
<td>5</td>
<td>1502'-'101</td>
<td>Different shades of black</td>
<td>Yes</td>
</tr>
<tr>
<td>10</td>
<td>2501'-'231; 2502'-'231; 2503'-'231</td>
<td>Yellow/Brown</td>
<td>No</td>
</tr>
<tr>
<td>14</td>
<td>n/a</td>
<td>Turquoise/White</td>
<td>No</td>
</tr>
<tr>
<td>23</td>
<td>1500'-'210</td>
<td>Turquoise/White</td>
<td>No</td>
</tr>
<tr>
<td>30</td>
<td>1000'-'230</td>
<td>Brown, non-colored</td>
<td>No</td>
</tr>
<tr>
<td>31</td>
<td>3301'-'231</td>
<td>Yellow/Brown</td>
<td>No</td>
</tr>
<tr>
<td>32</td>
<td>n/a</td>
<td>Yellow/Brown/Green</td>
<td>No</td>
</tr>
<tr>
<td>36</td>
<td>2201'-'100 ; 2202'-'100</td>
<td>Different shades of black</td>
<td>Yes</td>
</tr>
<tr>
<td>37</td>
<td>2101'-'110 ; 2102'-'110 ; 2103'-'110</td>
<td>Different shades of black</td>
<td>Yes</td>
</tr>
<tr>
<td>39</td>
<td>0801'-'100 ; 0802'-'100</td>
<td>Different shades of black</td>
<td>Yes</td>
</tr>
</tbody>
</table>

Table 4 Selected Seip Textiles

Due to the constraints of the glass plate housing, initial examination was limited to the obverse side of each textile. Later, the obverse and the reverse side of each of the selected textiles were examined more thoroughly under fluorescent white light to determine if the coloration was applied to both sides. Each glass case was opened and the textiles were carefully transferred to flocking paper (SPI Supplies). The obverse was observed and described. Different fragments that are housed together were examined and described separately. Although fabric structure was noted, it was not considered further since Thompson (2003) had already evaluated the structures of the Seip textiles. At this point, the obverse side of all textiles was photographed.

To be able to examine the reverse side of the textiles, they had to be turned over. Instead of doing this one piece at a time and compromising the integrity of these fragile textile fragments, another sheet of flocking paper was placed on top of the textile. At that
time, bottom and top flocking paper were held together firmly and flipped over in one motion, now displaying the reverse side of the textile fragments as they were placed in the housing. This reverse side was also observed, described and photographed.

The obverse side of all of the selected textiles was photographed in UV, photoflood, and simulated daylight conditions. The reverse side of all textiles was photographed with the Xenon flash (6420 K), and the photoflood lamp (3400 K) as light sources. However, to limit the textiles’ exposure to UV, the reverse side of only certain textiles was photographed in UV. When the results of the previous photography of the textiles’ obverse side showed patterns of coloration, then it was decided that further exposure was warranted.

**Acquisition of Samples**

The results of this photographic examination and documentation of the textiles were used to determine the location of the very small samples that were taken for further testing. Furthermore, particulate matter that had accumulated in the museum’s cases was collected.

**Particulate Collection**

With aging, textiles become very brittle and can fall apart into a dust-like material containing fiber fragments. At the time of moving the selected textiles from their housing onto the flocking paper, this particulate material that had accumulated within the glass housing cases was collected. When possible, the dust under differently colored sections of the individual textiles was collected separately even though contamination from one
color into the next could not be totally eliminated. This material was examined using optical microscopy (OM) as described in detail later.

**Tape Samples**

It was not feasible to use tape to examine the textiles for surface particulate. Due to their state of degradation and fragility, the textiles could not withstand this process and their integrity could not be guaranteed. Therefore, the non-fiber deposits, be they pigment or contaminant, found with the collected particulate dust or the specific yarn samples taken from the colored areas in the textiles had to be sufficient. Some but not all other non-fiber deposits could be identified. Certainly, it could be determined by observing under the optical microscope if the coloration in the fibers was correlated with these deposits, and if they were adhering to the fiber surfaces or occurred in the proximity of the colored fibers without otherwise being associated with them.

**Yarn Sample Acquisition**

The patterns detected by the photography, particularly UV, were taken into consideration when the samples and the sampling site on the textile fragments were selected. Furthermore, apparent degree of contamination by other materials such as soil was also considered during this process. Due to the great fragility of the textiles, and their limited quantity, not all eleven of the selected textiles were sampled for SEM-EDS analysis. Instead, samples were taken that were representative of the three textile groups that had been identified during the initial selection of textiles for further analysis: yellow/brown, white/turquoise and charred. Therefore only four of these eleven textiles were sampled for OM and SEM-EDS. These textiles were significantly different from
each other as prescribed by the initial grouping and yet, still contained all the features that
the whole group displayed. Therefore, samples were taken from textile numbers 4, 23, 36
and 37. Textile #4 represented the yellow/brown and textile #23 the turquoise on white
group. The charred textiles were represented by textile #36, which showed orange/red
and some blue coloration in UV light, and textile #37, which had enough blue coloration
to justify sampling. Textile #36 did not have enough blue area for sampling without
permanently altering the appearance of this textile.

<table>
<thead>
<tr>
<th>Textile # (Song)</th>
<th>Textile # (Thompson)</th>
<th>Apparent Color of the Fragments</th>
<th>Charred Yes/no</th>
<th>Samples Taken from colored in:</th>
</tr>
</thead>
<tbody>
<tr>
<td>4 0701'-'231</td>
<td>Yellow/Brown</td>
<td>No</td>
<td>Yellow/Brown/Green</td>
<td></td>
</tr>
<tr>
<td>23 1500'-'210</td>
<td>Turquoise/White</td>
<td>No</td>
<td>Shades of Turquoise/off-white/white</td>
<td></td>
</tr>
<tr>
<td>36 2201'-'100; 2202'-'100</td>
<td>Different shades of black</td>
<td>Yes</td>
<td>Orange/red, black</td>
<td></td>
</tr>
<tr>
<td>37 2101'-'110; 2102'-'110; 2103'-'110</td>
<td>Different shades of black</td>
<td>Yes</td>
<td>Blue</td>
<td></td>
</tr>
</tbody>
</table>

Table 5 Textiles Sampled for OM and SEM-EDS

The yarn samples that were taken directly from the textiles were chosen from the
apparently colored and apparently non-colored sections of the selected Seip Mound
textile fragments. Great care was taken to remove the smallest sample possible, which
also contained as much information as possible. Often that meant that the yarn samples
displayed several colors on the single pieces that were taken. These yarns or sometimes
just fibers were not any smaller than 3 mm but did not exceed 8 mm. The sample taking
was conducted in such a way that minimal damage occurred, and the removal did not leave a noticeable void in the textile fragment. In some cases, only fibers rather than complete yarns were removed. Surgical scissors (SPI Supplies) and very fine tipped tweezers were used to cut the material. The selected yarn samples were placed on an aluminum planchette covered with double sided carbon tape (8 X 20 mm, # 5072, SPI Supplies). The areas where these samples was removed from the textiles, and the lay-out of the samples on the planchettes were carefully drawn and documented for later analysis.

The sampling sites were documented on pictures previously taken and these were placed with the Ohio Historical Society as reference for future research. (Plate 1 Textile Sampling Sites).

Once in the lab, each yarn specimen was observed with a Bausch and Lomb macroscope under 3.18 X to 21.8 X magnification to determine if any encrustations or minerals were attached to the outside of the fibers. Then the samples were photographed at a 3.18 X, and divided further for the other examinations.

**Sub-sectioning of the Yarn Samples**

The initial plan of sub-sectioning each collected sample that was taken from the selected textiles was not feasible due to the great fragility of the fibers. Instead, while viewing the yarn samples under the Bausch and Lomb stereoscope, individual fibers were picked off the collected samples (on the planchettes) using fine tweezers and carefully placed onto microscope slides for further analysis.
**Microanalysis**

In analyzing the fibers at a microscopic level, the general fiber classes were identified (hair or bast). Furthermore, adherence of particles to the fibers, be they mineral colorant or soil was determined and when possible identified with the help of a particle atlas (McCrone and Delly, 1997). The previously created control mounts were used as comparative materials to the unknown mineral or pigment deposits that were observed in the artifact samples. Two pieces of equipment were used: the optical microscope and the scanning electron microscope.

**Optical Microscopy and Imaging**

Several slides were created for comparative purposes: lab grade hematite (Fe$_2$O$_3$) and copper sulfate (anhydrous and hydrated) were mounted on slides and used as controls to compare possible mineral deposits that may be found adhering to the fibers. Rabbit hair and milkweed that had been colored with an aqueous hematite solution and with an aqueous copper sulfate (blue vitriol) solution were also used for comparison. A few fibers removed from each simulated material were mounted in water (Refractive Index (RI) of 1.0), which is different from the fibers, and thus allows clear representation of the fiber surfaces. Additional fibers were mounted in Permount (RI of 1.55), which is similar to most fibers, and thus allows visualization of the fiber interiors. Using these techniques allows the determination of colorant penetration or adherence to the fibers.

The collected particulate matter of textiles and the yarn samples from each textile group were examined using optical microscopy. The particulate matter and few fibers removed from the yarn samples were examined using optical microscopy. This material
was mounted on microscope slides in water and Permount. With the aid of the Particle Atlas (McCrone and Delly 1997), the particles present were identified or classified if possible.

While this particulate represents the textiles as a whole and is a good source for information about the textile as a whole, it does not offer the possibility of the selective examination of the colors or patterns that indicate coloration as they had been identified by the photography. Therefore, it was still necessary to take samples from the individual colored or patterned areas within the textiles.

**SEM Imaging and EDS**

After having observed and photographed each yarn sample that was placed on carbon tape, these were carbon coated. At least one picture at a magnification between 400X and 3000X at an average of 10 keV was taken before the EDS data were collected (Appendix F). The EDS measurements were performed in the differently colored areas as they had been noted during the mounting of the fibers onto the planchettes.

Measurements were taken from: (1) several fibers of the same sample for average elemental composition, (2) at least two spots of a single fiber to compare elemental composition within one fiber, (3) material adhering to fibers to compare the elemental composition from fiber to adhering material. During the course of performing the SEM-EDS, it was noted that some particulate adhering to the fibers tended to charge up, while the fiber itself did not necessarily do so. Having particulate react differently than the fibers when placed under the electron beam indicates that the chemical signatures are different from each other.
ICP-MS

The SEM-EDS analysis produced a semi quantitative analysis of the elemental composition found in the samples. In spite of a number of elements present, organics were also present as was indicated by carbon peaks that were higher than the zero peaks in the EDS. Therefore the researcher decided to perform the Inductively Coupled Plasma Mass Spectrometry (ICP-MS) at a later time.

However, when the actual archaeological samples are analyzed, the terms of the digestion process must be modified to facilitate digestion of the elements that are present as determined by EDS in addition to the proteinaceous or cellulosic fibers that are to be digested. Nitric acid is a good digestion reagent, but depending on the mineral content, other acids may have to be incorporated into the process. These mixtures will also need to be incubated under pressure and with ultrasound in addition to the increased temperature in the water bath environment for total digestion to take place.
CHAPTER 5

RESULTS OF PROTOCOL APPLICATION: THE SEIP TEXTILES

In this chapter, the results of the examination of selected Hopewell textiles are summarized. All results are reported in terms of groups, and unless otherwise noted, the results were the same for each textile within the noted group.

These outcomes are summarized: (1) the visual and microanalysis (Appendix C, Tables 6, 7, 8, and 9), (2) EDS data averages and photography results (Appendix C, Table 10), (3) detailed EDS data (Appendix C, Table 11).

The Yellow/Brown Group

This group contained textiles designated Song (Appendix D) # 4, # 10 and # 31. There was a yarn sample taken and examined from # 4, while the particulate of the other textiles was examined in optical microscopy.

All of the textiles in this group were very fragile. Some of the textile fragments had possibly been glued to the glass housing. The colors of # 4 are reddish-brown, yellow-brown, and green, while # 10 and # 31 displays dark brown, reddish-brown and yellow colors. Under a nominal 2x magnification, the yarns were identified as 2-ply. The yarns displayed more than one color on the same yarn in some areas. These colors were distinct and sharply separated. The yarns appear totally saturated by color, but the
coloration is not level. Under greater magnification (20X), it could be determined that very small bits of brown fibers are mixed into the matrix of all three colored yarns. The estimated ratio is about 1:1000 or more, so they only occur very infrequently. The fibers themselves are fractured into very small pieces, even though the structure of the yarn is still intact. Furthermore, encrustations are easily recognized. Some of these are in the same shade of colors as the fibers, and appear almost specular in the light, indicating possible mineralization. However, there are few larger grains of whitish-blue deposit, much as a copper salt.

When photographing these textiles, their colors were best depicted in simulated daylight conditions. Adding the photoflood light still showed the colors albeit not as well. When comparing the colored patterns of the obverse and the reverse side, they were seen to match closely. They appeared almost identical. In one of the textiles, the colors were less sharply defined due to soil components or something similar, which covered the actual textile structure as well and filled in all the spaces between the yarns. When exposed to UV light, no fluorescence was achieved besides that of some degraded shell remains. No particular patterns were seen in UV reflected or fluorescence photography nor for IR exposure.

During the course of optical microscopy (Appendix E), rabbit hair fibers were identified, which is congruent with the findings by Song (1991). The medullae were ladder shaped and uni- or multi-serial. Some pigmentation is seen on cortical bridges, but not in all cases. Some scales were seen, but scale patterns were not observed. These rabbit hair fibers are stained in three colors in each textile: yellow, green and red-brown for # 4 and yellow, red-brown and dark brown for the two others. Many of these rabbit
hair fibers are fragmented and fractured. There were no apparent deposits of colorants on
the outside of the fibers as was determined in darkfield even though some encrustations
were observed. Therefore, the colors of the fibers did not correlate with those colors
observed in the rabbit hair. Some other mineral deposits not adhering to fibers were
observed. Furthermore, some unidentified bast fibers (Appendix E) were discovered.
Because their source has not been identified, these bast fibers will be referred to as the
“other” bast throughout this dissertation to differentiate these from milkweed and Indian
hemp bast fibers. Most of these fibers still have an outer layer attached much like a peel,
which is colored in shades of brown. In some cases, this outer layer is fractured, which
allows for the fibers beneath this covering to be seen. These fibers changed colors from
brown to bright yellow/orange in polarized light when the stage of the microscope was
turned by 45 degrees thus indicating that they were birefringent. This bast was not
restricted to any one color or one ply of the two-ply yarns, because all mounted fibers
from the dark-brown, red-brown, yellow and green yarns contained small amounts of this
bast mixed in with the rabbit hair.

**SEM-EDS**

Textile # 4 was sampled to perform SEM-EDS (Appendix F) as a representative
of the yellow/brown group. It was confirmed that these textiles contained hair fibers and
that some deposits adhered to the surface of the fibers. The brown fibers contained
organics as reflected by the quantity of carbon (C), but also materials which contained
copper (Cu) and small amounts of oxygen (O), iron (Fe), aluminum (Al), silicon (Si) and
phosphorous (P). Deposits on the brown fibers show the same spectra, but with lower
carbon peaks. The yellow and the green fibers also show the same spectra: a carbon peak higher than the baseline zero, a large amount of copper, some iron and negligible amounts of Al, Si, P, and sulfur (S). There was no difference in results between the yarns with two colors on their opposite ends that had been sampled or between yarns of one color that were analyzed later.

Special Features

In two out of the three textiles in this group special features were observed that had not been documented by previous researchers.

Textile # 10

When the textile fragments of # 10 were turned over to the reverse side, it became clear that at least three layers of fabric are folded on top of each other. It cannot be determined without unfolding all of these if the layers (Appendix D, Plates 13 and 14) belong to a single fabric with several designs and structures or if they belong to several different textiles. One of the fragments shows the degraded remains of leather. From this side, it does not seem as if the fabric fragments belonged to the same textile because of the obvious thread size differences. Colors differ from layer to layer, but also within the same fragment. Most strikingly, fringes or some sort of tassels or fringes are easily recognized on one piece. These fringes (Appendix D, Plate 13), made from intact complete 2-ply yarns are dark brown and they are attached to the dark brown section of this textile fragment, while the ends of the fringes rest loosely against the yellow colored part of the textile. None of the other fragments from textile # 10 show this sort of adornment. However, there is an area in the largest piece where unstructured dark brown
fibers cover the structured yellow surface of the underlying textile fragment. These fibers could also be the remains of fringes, but this can no longer be identified with confidence. Degraded bead or shell piece fragments are attached to the largest textile piece. Again, there is soil or sand contamination dispersed over the fragment surface.

**Textile #31**

When the textile fragments belonging to #31 were turned over to their reverse side, it revealed that two or more layers of fabric are folded on top of each other. The colored patterns seem to be the same. However, colored patterns and other fabric characteristics such as design features and structures cannot be determined indisputably without unfolding and examining all of these layers (Appendix D, Plate 14).

The colors observed on this side are dark brown, reddish-brown, and yellow. The colors differ from layer to layer, but also within the same fragment. The yarns are 2-ply and seem saturated by colorant. Most noticeably, fringes (Appendix D, Plate 14) were recognized on one piece. These fringes are composed of a complete yarn, i.e. a two-ply yarn that is plied in a low twist, while the two strands of the ply have a high twist. They are red-brown and hang against a dark-brown layer of fabric. The color is not level. It cannot be determined if these fringes are attached to the bottom fabric without unfolding both layers. Degraded bead or shell piece fragments are attached to the largest textile piece. It can no longer be determined if these remains had been sewn onto the fabric in the past. Again, there are soil or sand deposits on the textile structure, sometime filling in the spaces between yarns completely.
The Turquoise/White Group

This group contained the textiles designated Song (Appendix D) # 14 and # 23. A yarn sample was taken and examined from # 23, while the particulate of the other textile was examined.

Each of these textiles displays a turquoise pattern in different shades on an off-white background. Brighter white areas are contained within these turquoise motifs. These areas are somewhat degraded on the border between the turquoise and the white creating small voids. Furthermore, these pieces have degraded into a particular shape. The yarns are 2-ply, the fabric structure is very fine and the same for both textiles, creating a very thin, sheer fabric. The reverse side of each of the fragments is a mirror image of the obverse side with both sides saturated in color. However, each looks as if it could have been painted, because the colorants appear as a thick band located on top of the yarns. The areas of yarn that are covered by other yarns do not show any color. Both textiles have a small area that is folded.

In the photography, each piece was easily seen in any of the different lighting conditions. When exposed to UV light, fluorescence was achieved and the patterns that were demonstrated on film in simulated daylight conditions could clearly be seen in reflected or fluorescence UV photography. However, the lighting conditions outside of the visible range exposed patterns within the motif which could not be seen otherwise. The paint or dye in these different areas that appeared to be the same in daylight, showed different patterns in each of the different lighting conditions, indicating differences in reflectance and absorption of the light in these sections which points toward a different
chemical signature in these areas. Thereby different aspects of the colored pattern that had been applied to the fabric were revealed.

In optical microscopy (Appendix E), the materials from these textiles showed bast fibers (Plate 12) with strong polarization colors. Using darkfield, blue granules (Plate 12) attached to some of the fibers were found, while other fibers did not have these and were clean on the surface. There were some reddish mineral deposits on the slide and some encrustations on the outside of the fibers.

**SEM-EDS**

This group contained the textiles designated Song # 14 and # 23, from which the latter was sampled for EDS (Appendix F). During the first EDS, averages of entire fibers were measured. This did not establish a definite pattern of elemental composition differences between the colors. All the fibers except one contained variations of the same elements, Al, Si, S, Ca with high amounts of copper. Only the dark turquoise contained high relative quantities of copper, some sulfur and low amounts of silica and calcium. The white contained a lower amount of copper, but more silica and other elements Al, chlorine (Cl), potassium (K), Ca.

In the single fiber analysis, the two white materials were analyzed. The white fiber had a very high carbon peak, and a lower amount of oxygen and very low amounts of Cu, Al, Si, S, while the deposit on these fibers had a very high carbon peak, but also high copper and chlorine with small amounts of silica and sulfur. Most deposits were amorphous but some had crystalline structures. These had the same elemental composition, but again with a lower carbon peak than that of the fibers.
The Charred Group

This group contained the textiles designated Song (Appendix D) # 5, # 36, # 37 and # 39. A yarn sample was taken from all of the textiles except # 39 and examined, but the sampling was limited to particular colors that were revealed in the photography of these textiles.

All of the textiles in this group clearly show patterns in different shades of black, but also a hint of grey-white and dark blue. All of these patterns display an ovate motif, almost eye-like. Whether or not the yarns are saturated by colorant cannot be determined due to the extensive charring. Observed under a magnification of 2X to 20X, iridescent colors in blue, silver and shades of yellow to orange were observed on the surface of the fibers (Appendix E, Plate 18). Some encrustations were also observed.

After being photographed with the flash (simulated daylight), the colors that were barely discernable to the observer during the visual examination became much more pronounced, i.e. the two shades of black are actually seen as shades of grey. Additionally, a brownish-red can be seen, which was not detected with the unaided eye. A pattern of blue and an ochre-red stands out distinctly showing ovate shapes, some in an elliptical shape resembling an eye. Adding the photoflood light deepens the colorant differences. When comparing the colors and locations of color on the obverse and the reverse side, they are very similar, but not identical. Some of the pieces are folded.

When exposed to UV light, some fluorescence was achieved and the patterns that were demonstrated on film in daylight conditions were also seen in reflected or fluorescence UV photography. Fluorescence was not very strong, but changed in
intensity when the angle of the UV source was altered. The latter images do not display the patterns as clearly as under visible light, neither do the IR images.

When examined in optical microscopy (Appendix E), large, flattened black fiber bundles (Plate 9), which were not well separated, were found. Besides black rods, some having reddish edges, not much detail could be seen in brightfield. Internal structures could not be determined due to the severe charring of the fibers. Many of these blackened fibers were fractured. Surface details could be seen in darkfield and bast fibers were identified, many of which had cracks on the surface. The fibers were flattened and showed a shiny, graphite-like surface much as a piece of wood that had been burned. There were some reddish deposits on the outside of the fibers, and some mineral deposits not associated with the fibers were observed.

SEM-EDS

This group contained the textiles designated (Appendix F) Song # 5, # 36, # 37 and # 39. A yarn sample was taken from all of the textiles except # 39 and examined, but the sampling was limited to particular colors that were revealed by the photography of these textiles. The colorants adhere to the surface of the charred fibers. Whether or not colorant is within the fibers could not be established by optical microscopy (OM), and therefore the SEM-EDS became very important.

The first EDS analysis was done on the yarns that had been sampled and showed at least two colors on each. The results of this analysis showed small amounts of magnesium (Mg), Al, Si, P in all of the colored samples. The only variation found here
were the amounts of calcium and iron. However, a correlation between colors and the elemental composition could not be established.

Since these yarns displayed several colors and fell apart when they were placed on the planchette, and were possibly contaminated, #37 was also sampled. Only two single fibers with orange/red on one end and blue on the other end of the fibers were removed, and carefully placed on the planchette. Furthermore, several fibers were removed that contained no color at all as a control. Some of the orange/red material containing no fiber material was also sampled.

The elemental spectra showed a medium size peak of iron for the orange material without the fibers, followed by O, some Ca and Si, and minor amounts of C, K, Mg, Al, P. The fibers with the orange/red deposits showed basically the same spectra, but more oxygen and no potassium. The fibers with blue colorants showed the same spectra but higher peaks for calcium and carbon, and lower oxygen. The fiber that did not display colorants at all, had much higher carbon and calcium, and lower iron peaks, but also small amounts of Mg, Al, Si.

**The Composite Group**

This group was created during the examinations due to the complexity of these textiles, which was not revealed when only the obverse side was examined. Textiles #30 (Plate 8) and #32 (Plate 10) were identified as composite. #30 had been previously selected as the “non-colored” textile, while #32 had originally been part of the yellow/brown group. Only the particulate matter from these textiles was available for
microscopic examination and SEM-EDS was not performed. Due to the intricacy of these two textiles, further research as separate project is recommended.

Textile # 30 (Plates 8 and 14)

This textile has a relatively coarse structure especially when comparing it to # 14 or # 23. It is brown-beige on the obverse side with possible narrow brown stripes. These are very faint. There are some small green spots on the surface, probably copper salt residue, but they do not occur in a particular pattern. This textile had been glued to one side of the glass housing, and had to be prodded loose. The glue remains were collected to be analyzed later.

When this textile was turned to the reverse side, it became apparent that it was not a single textile, but rather three significantly different layers on top of each other. With the brown-beige textile layer that was examined earlier placed on the bottom, it looks like the least degraded component when compared to the others. The middle layer is severely degraded and could be skin or leather or some other similar substance; however, this must be addressed in further research. It almost looks like evenly applied “soil” with a cracked surface where the top portion is decorated with an evenly applied wide stripe (~6-8 cm) of white paint across from one side to the other, while the bottom portion is colored in a dark brown. Embedded into this middle layer is another darker brown, but widely spaced structure of yarns (Plate 14). This is almost net-like, and the yarns are definitely recognizable in a few small areas. This layer is severely degraded. Due to this degradation, it cannot be determined if these yarns are plied or not. Some rootlets are also seen as an intrusive feature on this side (reverse) to the right.
During the photographic examination, the obverse side of this piece did not display any particular features besides the brown-beige color of the whole piece. Any light source worked well to document the textile. However, when comparing the obverse and the reverse side, there are great differences as was described above, and adding the photoflood light as light source brings out the layering and the color differences better than simulated daylight conditions. When exposed to UV light, the obverse side of the textile did not show any differences from that observed under daylight conditions. Some of the glue residue fluoresced but there was no fluorescence from the textile. However, the reverse side of the textile showed intense fluorescence, particularly by the white stripe. Also, there may be some small, otherwise non-visible, white paint or glue remains that fluoresced on the bottom part of the otherwise brown areas. Only sampling these would clarify what the substance might be. The same patterns that were observed in daylight could be seen in IR without significant improvement.

Only the particulate matter recovered from beneath this textile was available for microscopic examination. Bast fibers were identified. Very few single fibers are seen, most occurred as fiber bundles. Some surface folding on the fibers could be identified. All of the fibers are colored in yellow to a brown or dark-red, sometimes more intensely in the middle than on the outside. Some small red and yellow granules are attached to the fiber surfaces. These occur also without association to the fibers. In polarized light, the fibers change from bright orange to brown and back to orange every 45° when the stage of the microscope is turned.
Textile Song # 32 (Plate 10)

The housing of textile Number 32 contained 16, possibly 17 textile fragments. Yellow, red-brown and dark brown areas are recognized much as in the other textiles from the yellow/brown group. Several green stained areas do not seem to be an integral part of the yellow, red-brown and dark brown colored pattern. This suggests that these textile fragments have been in contact with a copper object. Some of the textile structure is visible even though much of it is covered by soil or sand that is attached to the fragments. A few degraded shell remains are visible.

When the textile fragments were turned over to their reverse side, it is clear that two or more layers of fabric are folded on top of each other. The obverse side, placed as the bottom layer, is a textile structure made from yarns; the middle layer is made of leather or skin. Several layers (Plate 13) within this potential skin layer are visible in the small areas where chunks of this stratum are missing. All of this is covered in a yellow ochre colored mud-like layer, which has matting embedded within it. Some ends of the mat component stick out from this muddy layer indicating that this matting appears to have been constructed of some sort of grass or sedge, which cannot be further identified here. The structure of the matting also cannot be identified without destroying the piece. Furthermore, some bark or wood remains are also embedded in this stratum.

The colored patterns are very different when observed from this obverse side, and very little of the yarn textile structure is visible. There are small green stains randomly distributed across the entire surface. Again, there does not seem to be a correlation between green copper stains (Plate 10) and colored patterns within the textile. One cylinder shaped shell or bone bead was found. There are white spots in some areas, which
were identified as shell remains that are severely degraded. These also occur in no
particular pattern. Additionally, two small graphite-like, vitreous chunks were found on
this reverse side. The material is unlike anything else associated with this textile or any
other textile, and could not be identified. The colored patterns of the obverse and the
reverse side cannot really be compared since they are obviously part of very different
materials and strata, and they are not associated with each other beyond being layered on
top of each other. No folding was recognized.

When photographed, the colors dark brown, reddish-brown, yellow are best
depicted in simulated daylight conditions. Adding the photoflood emphasized these
colors, especially the reds and yellow. When exposed to UV light, fluorescence was
achieved only by the shell or bead remains. The colored patterns were seen in UV
reflected or fluorescence as well as in IR photography, but were not significantly
improved when compared to daylight.

Optical microscopy of the particulate material recovered from beneath the textile
shows rabbit hair in yellow, green, and different shades of red or brown (Plate 15). The
green is the same as that of the copper stains on the surface of the textile. The rabbit hair
shows fractures on the surface, and many fibers are broken extensively. All of the rabbit
hair is colored and the medullae are uni-serial and multi-serial. There are lots of deposits
on the fibers, but there are also many fibers without deposits. These deposits do not
necessarily correlate with the color of the fibers. Darkfield shows both, clean fibers and
fibers with encrustations on the surface. An insect fragment (Plate 17) was found, but it
was not identified. Overall, this piece is very complex and deserving of an extensive
investigation.

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CHAPTER 6

DISCUSSION AND IMPLICATIONS

The results of this research encompass two broad areas: (1) the development of a protocol for the examination of archaeological textiles and (2) the application of the protocol to selected textiles of Seip Mound, Ohio. In this chapter, the efficacy of the chosen methods and the results of examining selected Hopewell textiles are discussed.

**The Protocol and its Implications for the Study of Archaeological Textiles**

The methods in the proposed protocol were not only reasonable, but excellent choices for examining archaeological textiles. However, the analytical methods needed to be adjusted to working with a limited amount of material of unknown composition. The protocol includes a presentation of the sequence of methods to minimize invasive sampling of archaeological textiles, and to keep the samples as small as possible. To successfully sample archaeological textiles in a purposive and selective manner, the way in which sampling is done must be maximized, i.e. samples should be taken only from those regions on the textile that indicate differences in chemical signature. Furthermore, the methods must be adjusted to accommodate the small sample sizes and component concentrations. It must always be remembered that the materials dictate how one can or should proceed with the examinations, and that time and timing is very important.
Selection, Samples and Sequence

A great deal of information can be gained from a visual examination that is done with painstaking attention to detail. The initial visual examinations may have to be done in less than ideal conditions, but still can give many clues as to what to expect in the research that is to follow.

Particulate Matter and Tape Samples

During the first examination of the Seip textiles, it was recognized that there seemed to be a lot of particulate matter in the glass cases holding the textiles. The potential value of this material was recognized, and it was therefore collected when these cases were opened. Through examination it was learned that this material included degraded textile fibers mixed with contaminating matter. These fragments were more informative than had been expected; and if found in archaeological materials, it should be considered the first bit of information about the textiles’ state of degradation. The more particulate matter there is, the more likely the fibers will be severely fragile, which must be taken into account when choices are made about proceeding in the research.

Furthermore, since this material contains the severely degraded bits and pieces from the textiles, it can also be used (a) to assess the condition of the fibers, (b) to identify the fibers used in the continuous textile, and (c) to identify the coloration in general. It may even be possible to use this particulate for some of the other analytical methods besides OM such as micro infrared or Raman spectroscopy or even ICP-MS, because these require the sample materials to be ground up anyway and certainly would be appropriate as a first trial.
However, while collecting and using this particulate matter to study the textile as a whole may be useful, it cannot totally replace sample taking. The particulate cannot be associated directly with a particular area of the textile. It is also possible that the particulate is contaminated with material that is not from the textile. This is especially the case with textiles kept in boxes rather than those sealed between two glass plates for extended periods of time. Therefore, when studying coloration, selective sampling of particular colors or areas with different chemical signatures will still need to be done. Thereby, materials of different composition or color are kept separated and each can be analyzed by itself.

In the initial research design, surface particulate was to be collected in the form of tape samples. This would have given details about the contaminations the textile picked up over the years or even about some of the pigment and binder combinations that may have been used as paint. However, a severely mineralized and degraded textile will not survive the process of taking the tape sample. Therefore, tape samples were not taken from the Seip textiles. The material must dictate what can or cannot be done to avoid jeopardizing the artifacts.

_Lighting and Specimen Selection_

When selecting the specimen and sub-sampling from them, the lighting should be controlled. As shown in the forensic photography, different lighting conditions will reveal or hide details and this can be used to one’s advantage. However, differences in lighting should be taken into consideration before the initial visual examination and long before pictures are taken. The ideal lighting will allow the researcher to see as much as possible for the initial selection of the suitable textile specimen. Creating the ideal
environment to do the initial selection of textiles may permit fewer textiles to be selected to represent an assemblage, and thereby the sampling to be kept at a minimum. After working with several different light sources in this research, it became apparent that the fluorescent white light that was present at OHS when the initial specimen selection took place was not sufficient to take full advantage of the visual examination.

After pictures were taken in simulated daylight, many of the colored patterns on the textiles and the colors themselves became much more visible than when they were initially examined in fluorescent white light. As an alternative to the light sources that are available in the curation facilities, another light source that will reveal colors more effectively should be brought in. A small lamp creating simulated daylight (6500 K or D 65) combined with the photoflood lamp that is needed for the photography would do this adequately and allow for a more differential pre-selection from the total textile assemblage, increasing the efficiency of the initial textile selection, and thereby decreasing the number of textiles needed for a study. In controlling the lighting, more details in coloration are revealed and fewer textiles need to be sampled to capture these details. Otherwise, many details that may be important can potentially be overlooked. On the other hand, details may be recognized in a particular light, but may no longer be visible if the lighting conditions change, consequently creating confounding variables that could potentially alter reproducibility of the process.

Therefore, if the environment, i.e. the lighting, is standardized, fewer specimen removed could be more representative of the archaeological textile assemblage, thus facilitating an even more purposive selection process.
Lighting, Magnification and Sampling

The lighting employed during the initial evaluation and selection of the textiles is very important, but so is the continued availability of ideal lighting conditions during the actual sample taking. The photography may reveal details about colored patterns that may be difficult to sample if the lighting does not produce similar conditions. This can potentially increase the number of textile specimens and sub-samples unnecessarily.

Besides controlling lighting conditions, adequate magnification should be available during the work with the textiles. A nominal magnification of 8X was available before and during the actual sampling. Only later in the lab did it become apparent that the magnification could and should have been greater. The fibers within the yarn structures were very fragmented and mineralized, which was easily recognized under adequate magnification and the proper lighting conditions. This fragility explained the difficulties that occurred during sample taking when the pieces of yarns kept on falling apart as soon as they were touched with a pick or tweezers. Furthermore, the pictures on which the sample sites were documented should have been enlarged to ease the recording of minute details.

Besides extra time, patience, a steady hand and good color vision by the researcher, some flexibility to get usable samples is necessary even if that meant picking up single fibers that had only a second earlier been a yarn, but had fallen apart as they were moved. Hence, extra time must be allocated for the sampling process under magnification to make this task productive.
**Time and Flexibility**

A certain degree of flexibility must be built into the research design, because the actual conditions of the textiles and the contaminants are not known, and in the case of the Seip textiles could have been acquired over the course of 2000 years. This pertains but is not restricted to the application of the actual methods that were chosen, but also how they are conducted. This flexibility in the course of the research can only be facilitated by building in time to adjust and improvise methods and to overcome difficulties while retaining a certain willingness to let the data take the lead. The pursuit of clues that were not anticipated or were initially only thought to be minor is of the utmost importance; these could lead to greater knowledge about the materials.

For instance, the extensive time that was spent with the optical microscope examining the Seip materials was absolutely essential and could not have been replaced by more reading about the subject, or by any other test method or analysis. The same is true for the time spent with the macroscope. All materials degrade in their own way, and they have potentially encountered different environments, which makes this task particularly challenging, but also very exciting. Even if one has vast experience with this type of material, it is necessary to work through the material numerous times, and still be willing to go back to look again and again. The “other” bast fiber which has not been identified yet, was discovered this way. Initially, it was thought to be a contamination, since Song does not mention any kind of bast mixed with the rabbit hair in the yellow/brown group or any other textiles that she examined. However, after this “contamination” was found and occurred in other samples also, and the first slides were
examined repeatedly, it had to be concluded that Song had overlooked it in her research. Such a mistake is easily made when a feature only occurs a few times in a textile.

Furthermore, not only the fibers are to be examined, but also the particulate that is deposited on the fibers or is found in association with the fibers. Since there is not just morphology involved, but also optical properties, and any type of material could potentially be found given the history of the artifacts, this could turn into a separate, time consuming research. The importance of basic training in optical microscopy cannot be emphasized enough. Moreover, the types of controls, standards and other comparative materials can be planned and prepared, but they may have to be adjusted and improvised as any of the tests are conducted, all depending on what is encountered.

**Sequencing**

Besides time and flexibility, timing is also very important. The forensic photography as precursor to any sample acquisition forms the foundation of the protocol, and allows purposive sampling. However, during the course of working with the replicated material, it became apparent rather quickly that EDS is another key analysis that must always be performed to establish which elements to expect before attempting any quantitative elemental analysis on archaeological materials such as ICP-OES/MS, since the latter requires a digestion procedure. Depending on the elements present and their relative quantities that are detected in EDS, choice of digestion solute(s) and instrument may differ. It further became apparent that a successful trial run with replicated materials is essential to keep the sample size and the cost for instrument time at a minimum.
Elemental analysis was planned, because the literature clearly indicates that inorganic pigments, particularly ochre, had been used extensively by prehistoric Native Americans. Therefore, the inorganic path of analysis was prepared and conducted with the simulated materials containing ferrous oxide. This iron-containing material restricted the choices of which instrument to use. For OES, the sample size of the replicated materials that would represent an acceptable amount of material to be removed from the artifacts, was too small. Furthermore, certain elements cannot be measured with OES due to peak overlap. For example, calcium overlaps with argon on which the plasma is based. However, calcium was anticipated in artifacts that come from a limestone environment such as the Seip materials. So, MS was selected. However, the quadrupole instrument did not have the needed resolution, again, because the sample size was so small. Therefore, the more sensitive and expensive instrument with a double focusing detector had to be used, which had the resolution for a small sample analysis.

The samples had given no indication that the digestion was not a complete digestion, because with the unaided eye, sample materials could no longer be detected, and yet, the tubing kept clogging. The digesting process needs to be adjusted perhaps by prolonging incubation time, choosing higher temperatures, and using ultrasound. If the problem continues, a different acid or a combination of acids will be necessary to complete the digestion process. Experimentation with the replicates revealed sample preparation problems that need to be addressed further prior to the use of ICP-MS for archaeological textile analysis. Had the actual samples been used, much of them would have been wasted.
Two lessons must be learned from this experience. Before working with the actual artifacts, a set of replicated materials must be used and a successful trial run using “the method” be that ICP-OES/MS, GC-MS or any other analytical methods, must be achieved, so the methods of preparation can be adjusted properly. To facilitate this, appropriate materials must be replicated, which might mean plant or mineral collections, dyeing or painting and comparative standards must be created, so the unknown can be compared to the known. For many of the Old World dye plants these standards already exist. However, for North American dye plants comparative collections are in the early phases and subsequent analysis of colorant constituents have not yet been conducted (Jakes, Sibley and Yerkes 1994; Jakes 1996).

Furthermore, when working with the actual artifacts, a qualitative elemental analysis with EDS must be done before choosing ICP-OES/MS, so the methods of preparation can be adjusted to the mineral content, total digestion can be achieved, and the proper instrument can be chosen. Obviously, this pertains to the methods that will have to be tested and refined for the organic analysis as well.

**Limitations**

The degree of the fiber degradation and mineralization of the Seip materials was not foreseen. It caused frustrations during sampling, but also limited the degree of visual examination that was ethically justifiable without destruction of the textiles.

After the textiles were turned over, several layers of fabric, fringed fabric, a combination of fabric adorned with leather and two composite pieces were identified but these could not be separated. However, to truly examine these details, textiles need to be unfolded or even taken apart in the case of the leather decoration or the composite textiles.
to describe the structures underneath or to investigate the method of attaching the fringe or leather decorations. Due to the great fragility caused by the mineralization, these fibers did not retain the flexibility of non-degraded textiles to maintain their structure. With just a little bit too much manipulation of the material, it would have fallen apart into dust. This restricted the researcher. Therefore, the visual examination was not conducted to the same extent as if the materials were less degraded and fragile. Furthermore, in spite of the high degree of mineralization, the presence of organic compounds is indicated by the results of the EDS. The presence of organic components could not be disregarded, although mineralization does not preclude the existence of remaining organic materials in textiles in addition to copper possibly having formed malachite, a copper carbonate. For that reason, it was decided that the semi-quantitative elemental analysis done with EDS would be sufficient at this time, and the organic path would need to be explored more thoroughly before trace element analysis of the inorganic components. Since comparative materials and the analyses of colorant components need to be done prior to this pursuit, the sampling and analysis of Seip materials must be postponed.

The lesson to be learned from this however is an essential one in this type of work. The material must dictate the sequence of events—what, where, when and how.

The statistical implications of purposive sampling are not addressed here. Rather, the protocol has focused on qualitative analysis with minimum sample sizes, that is minimally destructive sample acquisition, in spite of having employed or suggesting to employ quantitative methodology.
The Seip Textiles: Materials, Structure and Decorations

Based on the results of the research, some inferences about the Seip textiles can be made. The construction of these textiles was far more intricate than previously thought, their design was more complicated, the colors were not just painted and they were exposed to copper somehow.

Fragments

The Seip textiles are highly fragmented. Although some fragments were curated in the same glass cases, their inclusion into the same case does not necessarily mean that they belonged to the same textile. However, since most field notes are not available anymore, it cannot be established which fragment belonged to a particular textile or even to a particular burial in most cases. It can be easily recognized that in the case of the charred textiles, the degradation environment or at least some of the treatments the textiles received, were similar. They were in a specific contact with fire: hot enough to cause charring, but not hot or close enough to cause total combustion. However, looking at such details as construction technique, style and possibly chemical composition some inferences can be made. For instance, the three yellow/brown textiles that were selected as representatives for the yellow/brown group had more details in common than just the coloration, even though that must be considered the most obvious one. Coloration on the yarns suggests different degrees of level colorant application in all three textiles. All fragments are constructed in the same twining technique. All yarns are 2-ply yarns. Based on these commonalities, the fragments could have been originally from a single larger
piece. Furthermore, # 10 and # 31 display the same type of adornment in form of fringes. In both textiles they are dark brown, 2-ply intact yarns.

**Colors and their Application**

The colors that were found in this research were surprising. With the large degree of ochre use documented in the literature, mineral pigments were thought to have been the source for the coloration of the Hopewell textiles.

Earthy colors were somehow expected, but blues and iridescent colors were not. Earthy colors were used indeed: yellow and green, red-brown and dark brown. However, these colors were inside clean fibers indicating that a dye must have been used as the colorant source. Otherwise, deposits beyond degradation products or contaminants should have been found on the fiber surfaces. Furthermore, the high carbon content shown in the EDS may be indicating the use of dye.

While it is possible that the yellow and green colors were each dyed with the same colorant, but the textile had different dye uptake and thereby produced yellow and green, it also could be that two different dyes were used, or the same dye with different dyeing aids. The same is true for the red-brown and brown. It still must be emphasized though that dyes must have produced these colors due to the clean fiber surfaces and the thorough coloration of the fiber interiors. Furthermore, these colors had to be applied by a technique that was not painting. Some resist technique must have been used to apply these colors, much as some of the early researchers believed when they suggested that the colors had been applied by either stamping or a resist technique. The only other textiles
that are known to have this application technique used are from Spiro, a Mississippian
temple mound that dates to almost 1000 years later.

Some of the green staining such as in # 32 did not correlate with the other colors.
It is therefore thought that these green stains are copper degradation products from direct
exposure to the copper, which was not noted in excavation reports, but is certainly
possible.

**Design and Construction**

The textile designated Song # 10 turned out to be not only folded several times
over, but also shows different colors within one layer and two or more colors on one
yarn. It also displays unusual adornments besides the coloration. For instance, the fringes
were not expected and had never been described before for the Hopewell. They are
attached to a brown area of one fragment and hang loosely over a yellow colored area.
Not only are different design elements used, but also the color differences were used to
maximize the effect. Furthermore, on one of the fragments, a piece of degraded leather
was discovered. This also had been attached to a piece with two or more colors. This
leather was not needed to uphold the structure of the textile, because the underlying layer
of fabric already performed that function. How exactly it was attached is unknown
because it could not be investigated without unfolding the two or more components and
would have caused the destruction of this textile fragment. However, it looks like it was
some sort of leather appliqué, sewn on top of an already colorful textile.

Furthermore, the textiles of the turquoise/white group are very fine and sheer,
almost resembling sheer curtain material. They must have taken many hours to produce,
probably requiring that their producer be released from attending to other necessary tasks such as food procurement, which were likely performed by other members of their family, group or village. This points to elaborate Hopewell dress and textile technology, thereby indicating much more sophisticated craftsmanship than was previously thought. It also implies that some craft specialization may have been in place since many hours would go into the production of such fine textiles that were elaborately decorated. This however allows the ability of some members of the community or even region to produce textiles to be traded for goods and services from other communities or regions who might be interested in bartering for their goods. This could indicate the Hopewell had a more complex social system that was previously suggested.

Colors, Optical Behavior and Mineralization

Besides revealing differences of the obverse and reverse side of the textiles by photography, the use of the different lighting was designed to reveal otherwise invisible chemical signatures on these textiles and the elemental analysis either confirmed or rebuked these. The results were mixed in the three textile groups, and answered fewer questions than they created.

*The Yellow/Brown Textile Group*

In this group, copper was found. Some of the fibers had a lot of organic material but also some copper while some fibers had lots of copper and not as much organic material. The textiles are thought to have not been in contact with copper, since the textiles were not found in direct contact with copper artifacts, but that statement might have to be revised. However, the textiles were definitely exposed to copper even if that
happened in the form of corrosion products that contained copper salts. As copper that was placed into the mound at a strata near the one containing the textiles degraded, rain or groundwater could have transported these copper decomposition products to the textiles. There, the copper replaced organic constituents of the fibers over time, mineralizing the fibers, which accounts for their fractile nature. This however does not mean that all of the organics are replaced by copper. EDS revealed that the same fiber had high carbon peaks in one area and in another area the carbon count was much lower. Furthermore, for the most part, the deposits on the fiber surfaces had the same elemental composition as the fibers with few exceptions, which is another indicator that the minerals on the outside of the fibers are the same as on the fibers’ interior. It also indicates great variation in the degree of fiber mineralization, since the values differ greatly within the same fiber.

There was no significant difference in the elemental composition between the yellow, brown and green fibers. The only difference that existed between the fibers and the deposits adhering to the fibers was the amount of carbon. The deposits showed peaks of carbon that were lower than the zero peak, indicating that the deposits are primarily inorganic, while the fibers retain considerable amounts of organic components. Since there is a difference in color, and yet, there were no differences in heavy elements, it must be concluded that the differences in colors of the yellow/brown group are due to organic components, e.g. dyes. Many different plants could have been the source for these colorants. Flavonoid dyes from plants such as oaks (*Quercus* sp.), onions (*Allium* sp.), or yarrow (*Achilla* sp.) could have provided dyestuff for the color yellow. Yellow corn (*Zea* sp.) could have yielded yellow as well. Brown could have been produced by
quinones found in plants such as walnuts (*Juglans* sp.) and red from plants such as sorrel (*Rumex* sp.) or from bedstraw (*Galium* sp.) plants. Even pokeweed (*Phytolacca americana* L.), which yields an anthocyan dye can color a textile in a bright red.

**The Turquoise/White Textile Group**

In the turquoise/white group, all colorants were painted on the fabric, and the turquoise shades all contained some form of copper, which easily could have been a copper salt such as copper oxide or sulfate. The white in this group was likely created by kaolin or some other type of white clay since the EDS revealed elements, such as Al and Si, which are generally found in clays. Suitable clays were abundantly available in Ohio and during historic times these were exploited in the porcelain industry, making this a reasonable conclusion. Furthermore, the copper from the Great Lakes region was also easily obtainable and has been found in many archaeological sites.

Fluorescence may be stimulated by UV, but is seen in the visible spectrum. The differences in patterns in the three spectra as revealed by the forensic photography were the most significant of all the textiles. Different patterns were revealed by optical behavior in reflected and fluorescence UV and IR spectra indicating differences in chemistry. The elemental composition did not necessarily indicate differences in heavy elements, which only leave the chemical species to be responsible for the different optical behavior in the three spectra.

**The Charred Textile Group**

In contrast to the other textile groups which contained very high amounts of copper, the elemental composition found in the charred group indicates the use of some
type of iron-containing clay as source of color. There was no difference in the amount of iron in orange/red and blue areas. Since the orange/red and blue had undergone the same types of degradation processes, and the other elements only differ in minute amounts, it must be assumed that the iron-containing components are actually different chemical species. To analyze these, different analytical methods have to be employed in addition to elemental analysis. Minerals such as hematite for red and glauconite for blue could possibly been the source of these colorants. The iridescent color scheme could have been produced by peacock ore (Cu₅FeS₄). However, without further analyses, this cannot be determined and must be treated as conjecture.

In the areas thought to contain no color at all, the iron was much lower, but carbon was higher reflective of the carbonaceous char and possibly may be an indicator that there are still organic components from the fibers and/or applied colors left that have not been totally combusted. On the other hand, since clays, due to their chemical structure, tend to bond easily with organic components that could have been responsible for coloration differences, it is therefore possible that the differences in previously visible colors no longer exist due to the charring or other decomposition processes, but that the elevated carbon remains as a residual marker.

The Composite Textile Group

The results of the analysis thus far were unexpected. These textiles are very complex, indicating the use of different materials such as fabric and leather, but also an unidentified bast fiber that also occurred in some of the other textiles.

Furthermore, the remains of an insect found in # 32 could have been a contamination that occurred after the textiles were excavated and curated at OHS, which
according to their staff has had some problems with termites years ago. The insect remains have not been identified, hence, possible contamination after the recovery from the mound can neither be confirmed nor rebuked. However, if the insect was acquired in prehistory, it could indicate that the textile may have been in skin contact with a person. This, depending on the type of insect, could indicate if that person was dead or alive, since insects can have distinct preferences for hosts.
CHAPTER 7

CONCLUSION

A major section of this dissertation research has concentrated on the development of a protocol to study colorant applications on archaeological textiles even if these are no longer visible. The focus is on using non-destructive or minimally destructive methods to classify the colorants that were used prehistorically as inorganic/organic and pigment/dye. This protocol was then applied to selected textiles from Hopewellian Seip Mound Group in southern Ohio to test its effectiveness on actual artifacts.

The protocol consists of a succession of analytical methods that have been adapted to be used with small sample sizes. If these are sequenced properly, the efficacy of the protocol is further optimized, thereby maximizing the acquisition of critical data while minimizing the need for large amounts of sampling material, and thus preserving the integrity of the artifacts.

The methods used were forensic photography using different lighting conditions (simulated daylight, infrared and ultraviolet), optical and scanning electron microscopy with energy dispersive X-ray analysis (EDS), and inductively coupled plasma mass spectrometry (ICP-MS) for elemental analysis. All methods were first tested on replicated materials thereby establishing suitable parameters for the application to archaeological
textiles. During the course of working with the replicas, limitations of the analytical methods were discovered and addressed for their use on archaeological materials, i.e. a limited quantity of material with an unknown chemical composition. These materials have potentially undergone degradation processes and could have been exposed to a variety of contaminants, which all must be considered during the analysis. For example, the digestion of the sampled material for the ICP was refined and a more appropriate instrument was selected based on the results of working with the replicas.

To reach the goal of using a minimal amount of sampling material, it is essential that the series of steps within the protocol are performed in the suggested sequence. One step builds on the previous one with several key tasks that must be performed before continuing with the analysis.

First, a comprehensive and systematic visual examination of the textile fragments (obverse and reverse side) must be conducted. Much can be learned if this is done meticulously. For instance, many details that had not been expected were discovered when the textiles were turned to the reverse side. The lighting conditions must be controlled for this process to guarantee reproducibility. Otherwise, the results will differ as the lighting differs.

Then suitable textiles that represent types within an assemblage based on the results of the visual examination are selected. For instance, the Hopewell textiles were grouped by commonalities in color and physical condition such as charring. Magnification should be used if necessary so no details are overlooked while controlling the lighting again.
Next, non-destructive forensic photography is used as a precursor to all the other steps. Before any other analytical method can be employed, the photography of the textiles in different lighting conditions must be performed because it reveals different chemical signatures due to colorant/substrate interaction even if these are no longer visible. This optical behavior is used to discriminate areas of diverse chemistry that can be correlated to colorant application. Thereby, the photography facilitates selective sampling of these areas, while areas of like chemistry do not need to be sampled. Thus, purposive sampling enables focused stratified sampling, increasing the opportunities for critical data acquisition while decreasing the need for the copious sampling of the material.

At this point, particulate matter should be collected. This particulate matter could consist of small textile fiber fragments and contaminants, which gives the first indication to the researcher about the textiles’ state of degradation. The more particulate matter there is, the more likely the textiles are severely fragile due to degradation or mineralization. Furthermore, the particulate can give detailed information about the textile as a whole, and it can be used for optical microscopy and possibly other analyses that pertain to the continuous textile such as IR.

After that, a detailed macroscopic examination, which also should be done in controlled lighting conditions, must be performed. Information about the physical state of the fibers can be gained. For instance, some of the Seip textiles showed many fractured and fragments of fibers within a yarn structure that still appeared to be intact, therefore making it very fragile. Furthermore, the colorant penetration and levelness of color can be determined, and adhering particulate can be observed.
This should be followed by the sub-sectioning the samples to divide the materials for further analysis. Subsequently, optical microscopy (OM) of the sub-samples can be performed to reveal fiber morphology and optical behavior. Additionally, the particulate that was collected earlier should be studied. This process should not be hurried since it takes some time to get accustomed to the samples and to recognize what is important in these samples. Images of these micrographs should be collected, and if a digital camera is used, the colors that are seen on the screen should be adjusted as much as possible and matched with the colors seen in the microscope.

Next, scanning electron microscopy (SEM) on the sub-samples should be performed. One of the strengths of SEM is the ability to capture detailed surface morphology that may not otherwise be detected. Furthermore, the great magnification that can be achieved with SEM shows details such as degraded scales from hair fibers or even the medulla cells that otherwise cannot be detected by optical microscopy.

While collecting images with the SEM, energy dispersive x-ray analysis (EDS) of the fibers and all their components, i.e. fibers and particulate adhering to them should also be performed. The EDS only gives the relative ratio of elemental composition of fibers and adhering materials, which cannot replace quantitative analysis. However, EDS is a good qualitative method to detect elemental composition. EDS constitutes a key step that allows the evaluation of carbon compared to the zero baseline, hence indicating the presence or absence of organic compounds. If organic components are present, organic analysis methods should follow as the next step, while the inorganic path of analysis should be taken if inorganic constituents are present. Furthermore, the relative ratios of elements detected by EDS in different areas of one fiber can be compared to each other,
to other fibers or to the elemental content of the particulate adhering to the fibers. Thereby, EDS can give information about ratio of organic and heavy elements, presence of mineral based colorants, the degree and variability of fiber mineralization, and possible contaminations.

For the inorganic path of analysis, such methods as ICP-MS/OES or LA-ICP-MS can be used. For these analyses, the potential problems that may occur during the digestion process that prepares spectrometry samples to be analyzed with various potentially suitable instruments were explored. When dealing with archaeological textile materials, it must be assumed that the samples will not digest well and that sample size is very small, and therefore, appropriate adjustments must be made. Knowing the relative ratio of elements present in the samples from results of the EDS will ease this process greatly, because appropriate replicas can be created, and the most likely successful digestion agent can be chosen to perform the spectrometry. For the organic path of analysis, such methods as gas or liquid chromatography followed by mass spectrometry, Micro IR and Raman must be explored. It must be assumed that problems similar to those found when preparing the samples for ICP-MS will also be found when other methods such as chromatography are used. Therefore, a successful trial run of every analytical method with replicated materials must be conducted before using artifacts. Thereby, subsequent analyses of the artifact material are most likely to be successful without having to be repeated, thereby keeping the amount of sample that is needed will be kept at the absolute minimum.

Based on an initial visual examination, eleven Seip textiles were selected and divided into three main color groups: (1) yellow/brown, (2) turquoise/white, and (3)
charred. These are representative of textiles from the actual assemblage. An extensive, painstaking visual examination under controlled light and description of the selected textiles’ obverse and reverse sides was conducted. Then both sides of the selected textiles were photographed in UV, warm and cool visible, and IR lighting. Based on the findings of the forensic photography, purposive sampling of the artifacts was conducted. Although the sample sizes were small, they were representative of the studied textile assemblage.

The yellow/brown textiles showed some encrustations on the fiber surfaces and severe fragmentation of the fibers. The fabrics were constructed of rabbit hair with colorant saturated fibers, which indicates that dyes were used as colorant sources. There were some surface deposits, but these could not be linked to the colors of the fibers. Many of the colored fibers showed no deposits at all.

The elemental composition of the three colors from this group did not show any differences between the colors. All colors contained a large amount of copper, some iron and small amounts of soil minerals, but they also contained large amounts of carbon, and some sulfur indicating organic materials in the fibers. It was concluded that the organic constituents of the fibers had been partially replaced by copper. While these textiles were not reported to have been in contact with copper, they must have been saturated by copper corrosion products carried by ground water, i.e. they were near copper albeit not directly adjacent to it. The encrustations that were observed in the optical microscopy and the severe brittleness of the fibers support that statement.

The textiles belonging to the turquoise/white group were made of milkweed fibers that were painted with different pigments. These colorants had not penetrated into the fibers, but adhered to the fiber surface. Different lighting conditions during the
photography showed various dissimilar aspects of the patterns, indicating differences in chemical signatures, and thereby the colorants applied. The elemental analysis indicated large amounts of copper, and small amounts of other elements. It was concluded that the white color was likely kaolin and that some of the other colors had been mixed with the kaolin or some other type of clay. These textiles were relatively stable when comparing them to the state of degradation of those from the two other groups.

The charred textiles were extremely fragile. Patterns no longer visible in fluorescent white light were visible using photography, and the simulated daylight showed them best. Ovate motifs in blue, ochre color and different shades of grey were found. When magnified, it could not be determined if the colored fibers were penetrated by dyes, because they are too charred to transmit light. However, different inorganic particulates adhered to the outside of these colored fibers, and some of these deposits were iridescent. Large amounts of iron were found in these colored fibers, but also some copper. The orange/red substance without any fiber material showed the same spectra as did the fiber but with lesser carbon peak, thereby verifying that the fibers still contain some amount of organic material. Fibers without any colorant on them had less iron and higher carbon and calcium peaks.

Two textiles were identified as composite. Both consisted of combination of several layers of materials: fabric, leather and matting. Due to the complicated nature of these specimens, they were only described but could not be addressed otherwise.
Suggestions for Further Research

All research seems to create as many questions as it provided answers, and with that it provides room for more work. This research is no exception. Based on the findings of this study, these are some suggestions for further work.

1. For the digestion process to prepare samples for spectrometry, ultrasound needs to be applied to the nitric acid/sample mixture to achieve better digestion.
2. Different potential digestion solutions or a combination of these such as hydrochloric acid (HCL) and hydrofluoric acid (HF) should be explored. Since these can cause problems such as the matrix effect, they must be tested with replicated materials.
3. The phytochemistry of many plants that were used by native Americans has not been analyzed yet. Colorant constituents must be identified in such genera that are known to yield dyes such as the native *Indigofera* species.
4. Standards of North American dye plants and their colorants must be created for potentially applicable methods such as Infrared and Raman spectra.
5. The methods to detect organic dye constituents such as Micro-Raman, Micro-IR, GC-MS need to be explored, tested with replicated materials and then applied to actual artifacts
6. The compositional data reported herein should be explored as to which inorganic pigments could have rendered the color to the textiles.
7. Quantitative elemental analysis should be conducted to link the colorants from the textiles to potential color producing minerals.
8. Composite images from the pictures that were taken should be created, thereby creating a likeness of what the textile might have looked like in the past, but also to potentially differentiate and sequence tasks in the production process.

9. The research done by Song and Thompson on the structures of the Seip textiles should be correlated with the chemical analysis and microscopy from this research.

10. Trace element analysis of copper artifacts should be done and compared and compared to the copper content of the textiles.

11. With the discovery of a bast fiber that had not been identified before, new aspects of Seip material culture came to light. The bast needs to be identified.

12. The two textiles that were identified as composite herein need to be studied in a separate project.

13. The insect piece that was found should be identified, and further research should consider that prehistoric people may have had insect infestation.
BIBLIOGRAPHY

The Amethyst Gallery, Inc.

Anonymous

Anderson, D.C.

Andrew, S.R. and D. Eastop

Baldia, M.O.

Bement L.C., et al.

Berns, R.S.

Brose, D.S.

Brose, D.S. and N. Greber (Ed.)
1978 *Hopewell Archaeology.* Kent State University, Kent, Ohio
Brown D.M. et al. (Ed.)

Brown, J.A.


Boyd, J.

Caldwell, J.R.

Casselman, K.L.

Cassman, V.

Catlin, G. [1832]

Chen, H.L., K.A. Jakes, D.W. Foreman,

Chen, R. and K.A. Jakes

Church, F.

Curl, A.M. and K.A. Jakes

Dancey, W.S. and P.J. Pacheco (Ed.)
1997 *Ohio Hopewell Community Organization*. Kent State University, Kent, Ohio.

Deetz, J. and P.S. Deetz

Densmore, F.

Duke, J.

Ericksen, A.G. and K.A. Jakes


Fitting, J.E.

Frison, G.C. and B.A. Bradley
1980 *Folsom Tools and Technology at the Hanson Site, Wyoming*. University of New Mexico Press, Albuquerque, NM.
Frison, G.C. and D.J. Stafford  

Goldstein, J.I. et al.  

Greber, N.  
1976 *Within Ohio Hopewell: Analysis of Burial Patterns from Several Classic Sites*. PhD Dissertation Case Western University, Cleveland, Ohio.


Gremillion, K.J., K.A. Jakes and V. Wimberley  

Hall, A.R.  

Hayashi, K.  

Hinkle, K.A.  

Jakes, K.A.  

112

Jakes, K. A. and A.G. Ericksen,
2001 Prehistoric Use of Sumac and Bedstraw as Dye Plants in Eastern North America Southeastern Archaeology, 20(1).

Jakes, K.A., J.E. Katon and P.A. Martoglio

Jakes, K.A. and J.C. Mitchell

Jakes, K. A., Sibley, L. R., Yerkes, R.W.,

Jennings, J.D.

Jodry, M.A.

Johnston-Feller, R.

Kehoe, A.B.

King, E. and J.S. Gardner
Klein, C.

Konigsberg, L.W.

Kuttruff, J.T.

Lahren, L. and R. Bonnichsen

Lovejoy, C.O.

Lutton, A.
The Microscopic and Chemical Analysis Research Center (MARC) 5-9-2005 personal communication


Martoglio, P.A, K.A. Jakes and J.E. Katon

McCrone, W.C. and J.G. Delly

McCrone, W.C, L. B. Mc Crone and J.G. Delly


Mindat.org Database


MURR


Rodriguez-Alegria, E.

Ruhl K.C. and M.F. Seeman

Saltzman, M. et al.

Sall, C. A., M. N. Zedeño, and R. J. Speakman

Schweppe, H.

Shetrone, H. C.

Shetrone, H. C. and E.F. Greenman

Simek, J.F., J.D. Franklin and S.C. Sherwood

Sibley, L.R. and Jakes, K.A.,

Sibley, L.R. and K.A. Jakes
Sibley, L.R.; K.A. Jakes and M. Swinker

Song, C.A.

Song, C.A., Jakes, K.A. and R.W. Yerkes

Speakman, R. J.

Speakman, R. J. and H. Neff

Speakman, R. J., H. Neff, M. D. Glascock, and B. Higgins

Srinivasan, R. and K. A. Jakes

Stafford, M.D., et al.

Struver, S. and G.L. Howard
Squier, G. E. and E.H. Davis
1848 Ancient Monuments of the Mississippi Valley. Smithsonian Contributions to Knowledge No. 1, Smithsonian Institution, Washington D.C.

Thompson, A. J.
2003 Textiles as Indicators of Hopewellian Culture Burial Practices. PhD Dissertation. The Ohio State University, Columbus, Ohio.

Tomlinson, P.

USDA-NRCS Plants Database


Verhecken, A.

Webb, W.S. and C.E. Snow
1974 The Adena People. The University of Tennessee Press, Knoxville, TN.

Weinstein-Evron, M. and S. Ilani

White, E.P.

Williams, R.A. and G. Williams
Wilmsen, E.N. and F.H. Roberts  

Wilson, K.  

Willoughby, C.C.  

Woodward, S.L. and J.N. McDonald  

1986  *Indian Mounds of the Middle Ohio Valley*. The McDonald & Woodward Publishing Company, Newark, OH.

Wouters, J.  


Wouters, J. and N. Rosario-Chirinos  

Zhang, X and R. A. Laursen  
Cinnabar is the primary ore of mercury. Its color is scarlet red, and its chemical composition is mercury sulfide (HgS).

Chelate is a chemical compound composed of a metal ion and a chelating agent. (http://scifun.chem.wisc.edu/CHEMWEEK/Chelates/Chelates.html, accessed 5-4-2005)

Chelating agent is a substance whose molecules can form several bonds to a single metal ion. In other words, a chelating agent is a multidentate ligand. (http://scifun.chem.wisc.edu/CHEMWEEK/Chelates/Chelates.html, accessed 5-4-2005)

Chromatography: A series of related techniques for the separation of a mixture of compounds by their distribution between two phases. In gas-liquid chromatography the distribution is between a gaseous and a liquid phase. In column chromatography the distribution is between a liquid and a solid phase.

Chromophore is a chemical group that is capable of selective light absorption resulting in the coloration of certain organic compounds. (http://www.thefreedictionary.com/chromophore, accessed 5-4-2005)

Compound: a term used generally to indicate a definite combination of elements into a more complex structure (a molecule) but it is also applied to systems with non-stoichiometric proportions of elements.

Conjugation: a sequence of alternating double (or triple) and single bonds. [E.g. C=C-C=C and C=C-C=O. Conjugation can also be relayed by the participation of lone pairs of electrons or vacant orbitals.]

Dye is a colorant of organic source containing one or many constituents that can color fibers at a molecular level.

Dyestuffs are intensely colored compounds applied to a substrate. Colors are due to the absorption of light to give electronic transitions.

Excited state is the state of an atom, molecule or group when it has absorbed energy and become excited to a higher energy state as compared to the normal ground state. The excited state may be electronic, vibrational, rotational, etc.
**Fluorescence** is a process in which material absorbs electromagnetic flux of a particular wavelength and emits energy that can be observed in the visible light spectrum, but also sometimes in the UV and IR spectra.

**Galena** is a bluish gray mineral with metallic luster consisting of lead sulfide (PbS). It is the principal ore of lead.

**Graphite** is the native element carbon (C). It is a grey, soft substance with a luster and often occurs in association with calcite or mica.

**Glauconite** is a yellow-green, green or blue-green silicate of iron and potassium.

**Goethite** is hydrated iron oxide a FeO(OH). Color is yellow, brown, brownish red to black. It is sometimes tarnished with iridescent colors. It also exists in its polymorphic form as Lepidocrocite, γ FeOOH. x H₂O (yellow, red, reddish-brown).

**Ground state** is the lowest energy state of an atom, molecule or ion.

**Gypsum** is a white or grey hydrated calcium sulfate (CaSO₄-2(H₂O)). The color can also be shades of red, brown and yellow. [http://mineral.galleries.com/minerals/sulfates/gypsum/gypsum.htm](http://mineral.galleries.com/minerals/sulfates/gypsum/gypsum.htm), accessed 6-6-2005

**Hematite** is an iron oxide (Fe₂O₃) and has a dull to bright red color. Other names used are specularite and oligiste. It is commonly earthy, ocherous or it is mixed with clay or other impurities.

**Hopewell** is the name for an archaeological culture that occurred in the Middle Woodland Period.

**Hydrolysis:** the addition of the elements of water to a substance, often with the partition of the substance into two parts, such as in the hydrolysis of an ester to an acid and an alcohol.

**Infrared spectroscopy** is the study of the absorption of infrared light by substances. [Since this corresponds to vibrational (and some rotational) changes, infrared spectroscopy provides valuable information about the structure of a substance. Detailed correlation tables exist relating infrared bands (absorbances) to functional groups.]

**Ion** is an atom or group of atoms that has lost or gained one or more electrons to become a charged species.

**Kaolin** is fine white clay. Its main constituent is kaolinite, a white, but also pale yellow, green or blue mineral that is composed of a hydrous aluminum silicate (AL₂Si₂O₅ (OH)₄).

**Lepidocrocite**, γ FeOOH. x H₂O (yellow, red, reddish-brown), see Goethite.
Limonite is a general term for mixture of different hydrated iron oxides, mostly Goethite. Its color is yellow, orange, reddish brown or brownish black.

Magnetite (Fe₃O₄) is black with metallic luster. This is one of the most abundant iron oxides and the source for iron ore. It often coexists with limestone, hematite, pyrite and other sulfides.

Manganese ore is black or purple and its chemical composition is manganese oxide Hydroxide (MnO(OH)). The mineral often occurs with calcite or limonite.

Mass spectrometry is a form of spectrometry in which, generally, high energy electrons are bombarded onto a sample and this generates charged fragments of the parent substance; these ions are then focused by electrostatic and magnetic fields to give a spectrum of the charged fragments.

Material Culture encompasses the objects that any one cultural group produced and/or consumed.

Mississippian is a name of an archaeological culture of mound builders that inhabited the greater Mississippi drainage between AD 800 and AD 1400.

Molecule is the smallest particle of matter that can exist in a free state. In the case of ionic substances, such as sodium chloride, the molecule is considered as a pair of ions, NaCl.

Ochre is a yellow and/or red mineral that belongs to the oxide or hydroxide group.

Ochre can contain some form of iron. However, ochre does not necessarily contain iron but can include a variety metals. The largest mineral database lists a total of 17 different ochres of which only three contain iron (http://www.mindat.org/search.php?name=ochre, accessed 4-22-2005).

The term ochre is frequently used interchangeably for hematite (Fe₂O₃) without discrimination based on elemental composition.

In some literature, the term ochre refers specifically to iron-containing red minerals that have been altered by humans.

Oligomeric compounds are polymers consisting of less than five monomers.

Organic colorant is a compound containing carbon that is derived from living beings such as plants or animals.

Optical activity is the property of certain substances to rotate plane polarized light. [It is associated with asymmetry. Compounds that possess a chiral carbon atom of all the same
'handedness' will rotate plane polarized light. Isomers that rotate light in equal but opposite directions are sometimes called 'optical isomers', although the better term to use is 'enantiomers'.

**Oxidation** is a chemical process in which the proportion of electronegative substituents in a compound is increased, or the charge is made more positive, or the oxidation number is increased.

**Photochemical reaction** is a chemical reaction brought about by the action of light.

**Pigment** is a soluble, generally inorganic colorant source that is made up of relatively large molecules and has to be used with a binder (egg, oil, etc.) to attach itself to the surface of a substrate.

Sometimes an organic dye that is already within the fiber can form a complex with a metal salt to create a large molecule that traps this complex within the fibers. This combination of organic dye metal salt complex does not need a binder and is not attached to the fiber surface. This complex is also called a **pigment** in some literature.

**Substrate** is the material that is to be colored such as fibers or textiles.

**Sample** is that piece of material that is removed from the artifacts to be examined in the laboratory.

**Spectrometer** is an instrument that measures the spectrum of a sample. [For example a mass spectrometer]

**Ultraviolet light** is radiation of an energy range than that of visible light but lower than that of ionizing radiations such as X-rays. Many substances absorb ultraviolet light, leading to electronic excitation. [This process is useful both as a means for characterizing materials and for stimulating chemical reactions (photochemical reactions.).]
APPENDIX A

FIGURES
Figure 1 Seip Mound Group Location
http://terraservice.net/ugsfentry.aspx?T=2&S=12&Z=17&X=386&Y=5432&W=2,
accessed May 31, 2005
Figure 2 Textile Structures as drawn by Willoughby
Figure 3 Adena Tablet (Brown et al. 1992)
Figure 4 Geographical Distribution of Mound Sites (Brown et al. 1992)
Figure 5 Hopewell Artifact 1 (Brown et al. 1992)

Twenty inches long, this stylized serpent's head with a forked tongue is fashioned from copper hammered into a thin sheet. The piece came from the largest of the 40 mortuary mounds at the Hopewell farm.

Discovered in 1892 in a burial mound at the Hopewell farm in Ohio, this naturalistically rendered redhorse sucker, or buffalofish, may have been regarded as a symbol of the underworld. The farm's name became attached to the entire Hopewell culture.
Figure 6 Hopewell Artifact 2 (Brown et al. 1992)

Figure 7 Hopewell Artifact 3, Seip Mound, (Brown et al. 1992)
Figure 8  Map Seip Mound Group by Squier and Davis (1848)
Figure 9 Quinones

Benzoquinone  1,4-Naphthoquinone  Anthraquinone

Figure 10 Berberine Cation, CAS 2086-83-1,
Figure 11 Electromagnetic Spectrum (Williams and Williams 2005)

Figure 12 Reflected Ultraviolet (UV) Photography Set-Up (Williams and Williams 2005)
Figure 13 Ultraviolet (UV) Fluorescence Photography Set-Up (Williams and Williams 2005)

Figure 14 Infrared (IR) Photography Set-Up (Williams and Williams 2005)
Figure 15 Painted Testfabric
Figure 26 Proposed Sample Sectioning for Possible Examinations
Archaeological Textile

Selection of Specimen Textile for Intensive Study

Visual Exam to Detect Color

Photography to Detect Colored Patterns & Description of Obverse & Reverse

Purposive Sample Selection

Macroscopic Exam to Detect Deposits on Fiber Surfaces & Coloration Variation and Capture Images

Divide Samples

Optical Microscopy
Detect Color Penetration & Adherence of Surface Deposits

SEM-EDS
Organic/Inorganic?

Inorganic Path such as

Organic Path such as

ICP-MS or ICP-OES

Micro-IR or Raman, Molecular Spectroscopy GC-MS

Particulate Collection
APPENDIX B

C¹⁴ DATES
(Balda M.O. 2005)
<table>
<thead>
<tr>
<th>Sample</th>
<th>Calibrated Date</th>
</tr>
</thead>
<tbody>
<tr>
<td>UCLA-290</td>
<td>2050±140BP</td>
</tr>
<tr>
<td>M-1830</td>
<td>2040±120BP</td>
</tr>
<tr>
<td>C-139</td>
<td>2040±120BP</td>
</tr>
<tr>
<td>UCLA-243</td>
<td>2040±100BP</td>
</tr>
<tr>
<td>RL-1197</td>
<td>2040±120BP</td>
</tr>
<tr>
<td>DIC-472</td>
<td>2030±55BP</td>
</tr>
<tr>
<td>UGA-1581</td>
<td>2025±80BP</td>
</tr>
<tr>
<td>UGA-4641</td>
<td>2025±105BP</td>
</tr>
<tr>
<td>M-674</td>
<td>2020±150BP</td>
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<tr>
<td>Beta-68758</td>
<td>2010±60BP</td>
</tr>
<tr>
<td>Beta-6396</td>
<td>2000±70BP</td>
</tr>
<tr>
<td>UGA-1893</td>
<td>1995±150BP</td>
</tr>
<tr>
<td>Pit-1237</td>
<td>1990±35BP</td>
</tr>
<tr>
<td>DIC-6621C</td>
<td>1980±155BP</td>
</tr>
<tr>
<td>M-908</td>
<td>1975±200BP</td>
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<tr>
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<td>1970±30BP</td>
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<td>Beta-55270</td>
<td>1970±100BP</td>
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<td>Pit-1241</td>
<td>1965±50BP</td>
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<td>UGA-1550</td>
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<tr>
<td>Beta-1561</td>
<td>1960±90BP</td>
</tr>
<tr>
<td>Beta-55271</td>
<td>1960±90BP</td>
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<tr>
<td>GX-2536</td>
<td>1960±150BP</td>
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<tr>
<td>OWU-464</td>
<td>1955±125BP</td>
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<td>C-136</td>
<td>1951±200BP</td>
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<tr>
<td>DIC-1189</td>
<td>1950±18BP</td>
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<tr>
<td>OWU-154</td>
<td>1950±18BP</td>
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<tr>
<td>OWU-323</td>
<td>1950±18BP</td>
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<tr>
<td>Uga-2419</td>
<td>1950±55BP</td>
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<td>Beta-29967</td>
<td>1950±50BP</td>
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<td>GX-2537</td>
<td>1950±115BP</td>
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<td>Beta-5564</td>
<td>1949±120BP</td>
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<tr>
<td>UCLA-2428</td>
<td>1938±75BP</td>
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<td>DIC-2660</td>
<td>1930±50BP</td>
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<tr>
<td>DIC-2673</td>
<td>1920±320BP</td>
</tr>
<tr>
<td>UGA-2150</td>
<td>1910±60BP</td>
</tr>
<tr>
<td>Lab-?</td>
<td>1910±80BP</td>
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<tr>
<td>Lab-??</td>
<td>1900±50BP</td>
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<tr>
<td>UCLA-241</td>
<td>1900±100BP</td>
</tr>
<tr>
<td>DIC-801</td>
<td>1900±400BP</td>
</tr>
<tr>
<td>UCLA-292</td>
<td>1895±100BP</td>
</tr>
<tr>
<td>OWU-172</td>
<td>1890±100BP</td>
</tr>
<tr>
<td>M-650</td>
<td>1890±200BP</td>
</tr>
<tr>
<td>Beta-67234</td>
<td>1880±70BP</td>
</tr>
<tr>
<td>Pit-1238</td>
<td>1875±75BP</td>
</tr>
<tr>
<td>Beta-145868</td>
<td>1870±40BP</td>
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<td>Beta-29969</td>
<td>1870±50BP</td>
</tr>
<tr>
<td>UGA-4640</td>
<td></td>
</tr>
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APPENDIX C

TABLES
<table>
<thead>
<tr>
<th>Textile as Whole</th>
<th>Description and Colors</th>
<th>Textile/ Fragments</th>
<th>Visual</th>
<th>Macro</th>
<th>Optical Microscopy</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td># 4, several only 6 studied</td>
<td>Yellow-brown, red-brown, green; colored patterns on obverse &amp; reverse;</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td># 10, 11 fragments</td>
<td>Dark brown, red-brown, yellow on obverse &amp; reverse, and within same fragment; 2 or more colors per fragment, 3 or more layers w/different colors; similarity of patterns cannot be determined without unfolding, color</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td># 31, 7 fragments</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Yarns</th>
<th>Color</th>
<th># 4</th>
<th>more than one color on one yarn; yarn saturated, non-level</th>
<th>Red, yellow, green color inside the rabbit hair fibers</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td># 10</td>
<td>fringes made of dark brown 2-ply yarn attached to dk brown part of fragment, but hanging freely over yellow part; different yarn sizes; some dk brown fiber deposit on yellow</td>
<td>Dk brown, red-brown, yellow color inside rabbit hair fibers; bast brown</td>
</tr>
<tr>
<td></td>
<td></td>
<td># 31</td>
<td>fringes made of dark brown 2-ply yarn on top of dk brown frag., not clear if they are attached,</td>
<td>Colors as # 10, bast yellow</td>
</tr>
<tr>
<td>Yarns</td>
<td>Structure</td>
<td># 4</td>
<td>2-ply, very fragile</td>
<td>Rabbit Hair: very fractured, very fragmented, fibrillated, some scales; few bast, throughout textile: sometimes w/ peel, changes color in polarized light every 45 degrees</td>
</tr>
<tr>
<td></td>
<td></td>
<td># 10</td>
<td>n/d</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td># 31</td>
<td>n/d</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Yarns</th>
<th>Structure</th>
<th># 4</th>
<th>2-ply, very fragile</th>
<th>Rabbit Hair: very fractured, very fragmented, fibrillated, some scales; few bast, throughout textile: sometimes w/ peel, changes color in polarized light every 45 degrees</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td># 10</td>
<td>n/d</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td># 31</td>
<td>n/d</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Yarns</th>
<th>Deposit</th>
<th># 4</th>
<th>Soil, sand etc covering some of the textile structure</th>
<th>Encrustations; Some deposits in DF, do not correlate w/color</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td># 10</td>
<td>n/d</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td># 31</td>
<td>n/d</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Yarns</th>
<th>Non-adhering</th>
<th># 4</th>
<th>n/d</th>
<th>Light blue; white</th>
<th>2 or more different deposits, anisotropic</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td># 10</td>
<td>n/d</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td># 31</td>
<td>n/d</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Yarns</th>
<th>Other</th>
<th># 4</th>
<th>Glued to housing by curators</th>
<th>Small bits of brown fibers or peel worked in the matrix yarn</th>
<th>Unidentified bast fibers; not Milkweed or Indian hemp</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td># 10</td>
<td>n/d</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td># 31</td>
<td>n/d</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 6 Summary Yellow/Brown Textile
<table>
<thead>
<tr>
<th>Textile as Whole</th>
<th>Description and Colors</th>
<th>Visual</th>
<th>Macro</th>
<th>Optical Microscopy</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td># 14, 3</td>
<td>fragments similar in shape &amp; color; turquoise/white/off-white; white surrounded by void; one fragment folded, does not distort pattern; relatively clean from soiling</td>
<td>very sheer textile &amp; structure; colored pattern on obverse &amp; reverse</td>
<td>Bast fibers w/ strong polarization colors, looks like milkweed;</td>
</tr>
<tr>
<td></td>
<td># 23, 1</td>
<td>turquoise/ shades of green/white/off-white;</td>
<td></td>
<td>unidentified bast fiber w/ inclusions; yellow, brown</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Yarns</th>
<th>Color</th>
<th># 14</th>
<th>Yarns not saturated; color voids where other yarns crossed; turquoise washed out in some areas turning it into shades of green; more than one color on one yarn;</th>
<th>painted onto surface</th>
<th>No colors within fibers</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td># 23</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

| Structure | # 14 | 2-ply, very fine | n/d | | |
|           | # 23 | n/d | | | |

| Deposits | Adhering | # 14 | n/d | Encrustations | Blue granules, non-colored smaller; red, small |
|          | # 23 | n/d | | | |
| Non-adhering | # 14 | n/d | n/d | Mineral, isotropic |
|             | # 23 | n/d | | |

| Other | # 14 | n/d | n/d | n/d |
|       | # 23 | n/d | n/d | n/d |

Table 7 Summary Turquoise/White Textiles
<table>
<thead>
<tr>
<th>Textile Fragments</th>
<th>Visual</th>
<th>Macro</th>
<th>Optical Microscopy</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yarns</td>
<td>Color</td>
<td>Structure</td>
<td>Adhering</td>
</tr>
<tr>
<td># 5, 15 frag.</td>
<td>n/d</td>
<td>n/d</td>
<td>n/d</td>
</tr>
<tr>
<td></td>
<td>n/d</td>
<td>n/d</td>
<td>n/d</td>
</tr>
<tr>
<td></td>
<td>n/d</td>
<td>n/d</td>
<td>n/d</td>
</tr>
<tr>
<td></td>
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</tr>
<tr>
<td></td>
<td>n/d</td>
<td>n/d</td>
<td>n/d</td>
</tr>
</tbody>
</table>

Table 8: Summary of Charred Textiles

- **Textile as Whole**: Description and Colors
- **Charred**: Twined fabric structure same for all; pattern in different shades of black, blue, grey; ochre; pattern similar but not same on obverse & reverse; ovate or eye shaped motifs; some folding.
- **Color**: Saturation cannot be determined; ochre colored areas seem painted.
- **Fiber surface structure**: Cracked, non-colored areas graphite-like & shiny, looks like burned wood surface (DF); edges of fibers reddish; identified as bast; many fiber bundles.
- **Yarns Structure**: Difficult to determine, but seems 2-ply.
- **Adhering Deposits**: Encrustations; some colorants on surface; colors iridescent in yellow/orange and blue tones; Red deposits (DF); Some mineral
- **Non-adhering Deposits**: Some mineral
- **Other**: Due to charring, no internal structures visible; BF just shows black rods and some deposits; Textile fragments as a whole relatively stable, but yarns by itself very fragile.
<table>
<thead>
<tr>
<th>Textile as Whole</th>
<th>Description and Colors</th>
<th>Textile/ Fragments</th>
<th>Visual with some Magnification (loupe)</th>
<th>Optical Microscopy of Particulate</th>
</tr>
</thead>
<tbody>
<tr>
<td># 30, non-colored control; Three layers identified</td>
<td>obverse</td>
<td>Coarse structure; beige-brown; faint stripes (?); fragile</td>
<td>Unidentified bast fibers; few single fibers, mostly fiber bundles with some surface folds; fibers change colors every 45° in polarized light;</td>
<td></td>
</tr>
<tr>
<td></td>
<td>middle</td>
<td>unidentified, amorphous, dk. brown layer (soil-like) w/ cracked surface;</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>reverse</td>
<td>top painted w/ wide, white stripe from left to right; open, net-like textile embedded into middle layer;</td>
<td></td>
<td></td>
</tr>
<tr>
<td># 32, 16 fragments; thick, three or more layers identified</td>
<td>obverse</td>
<td>Large green stains from contact w/ copper; textile dark brown, red-brown &amp; yellow</td>
<td>Rabbit hair identified in yellow, green and different shades of red or brown; clean fibers &amp; fibers with deposits in DF, but not correlated to color of rabbit hair; Unidentified bast fibers present;</td>
<td></td>
</tr>
<tr>
<td></td>
<td>middle</td>
<td>Leather like structure, possibly two or more layers;</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>reverse</td>
<td>Some green stains, some really small like from degraded copper beads; textile structure only visible from obverse layer; Matting embedded into clay-like layer between middle and reverse. Mat material grass or sedge; some bark remains; vitreous, graphite-like, chunk, ~1 cm</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Yarns</td>
<td>Color</td>
<td># 30</td>
<td>Obverse beige-brown; reverse dark brown</td>
<td>Yellow, brown &amp; red</td>
</tr>
<tr>
<td></td>
<td></td>
<td># 32</td>
<td>Dark brown, red-brown, yellow; reverse n/d; colored pattern not alike due to layering; no correlation between colored pattern and green stains;</td>
<td></td>
</tr>
<tr>
<td>Structure</td>
<td># 30</td>
<td>obverse 2-ply; reverse cannot be determined</td>
<td>n/d</td>
<td></td>
</tr>
<tr>
<td></td>
<td># 32</td>
<td>2-ply for obverse; others non-yarn structures</td>
<td>n/d</td>
<td></td>
</tr>
<tr>
<td>Deposits</td>
<td>Adhering</td>
<td># 30</td>
<td>n/d</td>
<td>Small, red &amp; yellow granules;</td>
</tr>
<tr>
<td></td>
<td></td>
<td># 32</td>
<td>Soil etc caked into textile structure on obverse side</td>
<td>considerable</td>
</tr>
<tr>
<td>Non-adhering</td>
<td># 30</td>
<td>Green spots from copper salts</td>
<td>Small, red &amp; yellow granules; other isotropic minerals</td>
<td></td>
</tr>
<tr>
<td></td>
<td># 32</td>
<td>Degraded shell remains; covered w/soil etc</td>
<td>considerable</td>
<td></td>
</tr>
<tr>
<td>Other</td>
<td># 30</td>
<td>Had been glued to housing; requires further research</td>
<td>n/d</td>
<td></td>
</tr>
<tr>
<td></td>
<td># 32</td>
<td>Intrusive rootlets on reverse; fragment of unidentified insect; cylinder shaped shell or bone bead on reverse; requires further research</td>
<td>n/d</td>
<td></td>
</tr>
</tbody>
</table>

Table 9 Composite Textiles—Require Further Research
<table>
<thead>
<tr>
<th>Textile</th>
<th>Planchette Sample</th>
<th>Colored Sample</th>
<th>EDS Elemental Ratio (averages)</th>
<th>Effectiveness of Photography</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>1</td>
<td>Green</td>
<td>3 Al; 2 Si; 1 P; 6 Fe; 54 Cu</td>
<td>Simulated Daylight +++</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Photoflood ++</td>
</tr>
<tr>
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<td>Infrared +</td>
</tr>
<tr>
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<td></td>
<td></td>
<td></td>
<td>UV Reflected ++</td>
</tr>
<tr>
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<td></td>
<td></td>
<td></td>
<td>UV Fluorescence ++</td>
</tr>
<tr>
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<td></td>
<td></td>
<td></td>
<td>Shell remains ++++</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Yellow</td>
<td>5 Al; 5 Si; 4 S; 2 Fe; 54 Cu</td>
<td>UV fluorescence illustrates contaminants such as shell remains, dust, glue or tape residue best!</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Green</td>
<td>5 Al; 4 Si; 1 P; 6 S; 4 Fe; 47 Cu</td>
<td></td>
</tr>
<tr>
<td>2</td>
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<td>Yellow</td>
<td>6 Al; 3 Si; 1 P; 6 S; 5 Fe; 47 Cu</td>
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</tr>
<tr>
<td>3</td>
<td>Yellow</td>
<td>Brown</td>
<td>5 Al; 7 Si; 2 P; 3 S; 12 Fe; 39 Cu</td>
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</tr>
<tr>
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<td>5 Al; 3 Si; 1 P; 5 S; 5 Fe; 51 Cu</td>
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</tr>
<tr>
<td>23</td>
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<td>1 Si; 7 S; 2 Ca; 62 Cu</td>
<td>Simulated Daylight ++</td>
</tr>
<tr>
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<td></td>
<td>White</td>
<td>9 Al; 15 Si; 4 Cl; 4 K; 7 Ca; 26 Cu</td>
<td>Photoflood ++</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Infrared +++</td>
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<td></td>
<td></td>
<td></td>
<td>UV Fluorescence +++++</td>
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<tr>
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<td></td>
<td></td>
<td></td>
<td>UV Reflected ++++</td>
</tr>
<tr>
<td>6</td>
<td>Whitish</td>
<td>Green</td>
<td>1 Al; 1 Si; 7 S; 3 Ca; 59 Cu</td>
<td>Each type of lighting brings out different features of the patterns, implying a different chemical signature. These indicate different absorption, reflectance &amp; fluorescence at the specific wavelengths.</td>
</tr>
<tr>
<td>7a</td>
<td></td>
<td>Off-white</td>
<td>7 Al; 6 Si; 3 S; 4 Ca; 49 Cu</td>
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</tr>
<tr>
<td>7b</td>
<td>One yarn</td>
<td>Olive</td>
<td>2 Al; 3 Si; 5 S; 3 Ca; 58 Cu</td>
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</tr>
<tr>
<td></td>
<td></td>
<td>Whitish green</td>
<td>1 Al; 2 Si; 7 S; 2 Ca; 58 Cu</td>
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</tr>
<tr>
<td>8</td>
<td>One yarn</td>
<td>Dark turquoise</td>
<td>8 S; 1 Ca; 62 Cu</td>
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</tr>
<tr>
<td></td>
<td></td>
<td>Olive</td>
<td>4 Al; 7 Si; 2 S; 3 Ca; 54 Cu</td>
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</tr>
<tr>
<td></td>
<td></td>
<td>Off-white</td>
<td>5 Al; 5 Si; 4 S; 8 Ca; 46 Cu</td>
<td></td>
</tr>
<tr>
<td>36</td>
<td>Charred</td>
<td>Reddish</td>
<td>1 Al; 3 Si; 1 P; 9 Ca; 58 Fe</td>
<td>Simulated Daylight +++</td>
</tr>
<tr>
<td></td>
<td>One yarn</td>
<td>Blue/ metallic</td>
<td>1 Al; 3 Si; 1 P; 9 Ca; 58 Fe</td>
<td>Photoflood ++</td>
</tr>
<tr>
<td>37</td>
<td>Charred</td>
<td>Blue</td>
<td>2 Mg; 2 Al; 3 Si; 30 Ca; 35 Fe</td>
<td>Simulated Daylight +++</td>
</tr>
<tr>
<td></td>
<td></td>
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<td></td>
<td>Photoflood ++</td>
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<tr>
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<td>Infrared +</td>
</tr>
<tr>
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<td></td>
<td></td>
<td></td>
<td>UV Fluorescence ++</td>
</tr>
<tr>
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<td></td>
<td></td>
<td></td>
<td>UV Reflected ++</td>
</tr>
<tr>
<td>9a</td>
<td></td>
<td>Reddish</td>
<td>1 Al; 3 Si; 1 P; 9 Ca; 58 Fe</td>
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</tr>
<tr>
<td>9b</td>
<td></td>
<td>Blue</td>
<td>2 Mg; 2 Al; 3 Si; 30 Ca; 35 Fe</td>
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<tr>
<td>10</td>
<td>Brown/red/blue</td>
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<td>2 Mg; 2 Al; 3 Si; 45 Ca; 17 Fe</td>
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<tr>
<td>11a</td>
<td>White stripe</td>
<td>Blue</td>
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<td></td>
</tr>
<tr>
<td>11b</td>
<td>One yarn</td>
<td>Blue</td>
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</tr>
<tr>
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<td></td>
<td></td>
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<tr>
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<td></td>
<td></td>
<td>2 Mg ; 1 Al; 3 Si; 1P; 33 Ca; 30 Fe</td>
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</tr>
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</table>

Table 10 EDS Data Averages and Photography Results

Continued
Table 10 continued

<table>
<thead>
<tr>
<th>Textile</th>
<th>Planchette</th>
<th>Colored Sample</th>
<th>EDS</th>
<th>Comments Photography</th>
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<tbody>
<tr>
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<td>13</td>
<td>Red/brown</td>
<td>2 Mg ; 1 Al; 3 Si; 1 P; 33 Ca; 30 Fe</td>
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<tr>
<td></td>
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<td>Blue</td>
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<td>14b One yarn</td>
<td>White</td>
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<td></td>
<td></td>
<td>Blue-grey</td>
<td>4 Mg ; 2 Al; 4 Si; 1 P; 38 Ca; 21 Fe</td>
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</tbody>
</table>

Legend:
The use of ++ denotes the ranking of effectiveness for the photography from highest ++++ to the lowest +.

Note:
1) The elemental composition ratios are rounded here, while the exact EDS data can be found in Appendix F
<table>
<thead>
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<th>Element</th>
<th>Weight %</th>
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</thead>
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<td>0 0 0 0 0 0 0 0 0 0</td>
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<tr>
<td>Al</td>
<td>2.99 3.3 4.09 3.06 3.37 4.46 5.16 5.9 5.44 5.07</td>
</tr>
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<td>Si</td>
<td>1.66 1.25 3.38 1.99 2.43 4.54 4 2.99 3.16 6.47</td>
</tr>
<tr>
<td>P</td>
<td>2.04 0.84 0.94 0.74 0 0 0.99 1.32 0.71 1.71</td>
</tr>
<tr>
<td>S</td>
<td>4.83 4.66 5.17 5.56 4.31 4.36 6.28 5.79 4.57 3.22</td>
</tr>
<tr>
<td>Cl</td>
<td>0 0 0 0 0 0 0 0 0 0</td>
</tr>
<tr>
<td>K</td>
<td>0 0 0 0 0 0 0 0 0 0</td>
</tr>
<tr>
<td>Ca</td>
<td>0 0 0 0 0 0 0 0 0 0</td>
</tr>
<tr>
<td>Fe</td>
<td>8.77 2.55 4.14 6.71 6.48 2.26 4.12 4.49 5.14 12.18</td>
</tr>
<tr>
<td>Cu</td>
<td>50.15 59.31 51.64 52.54 55.38 54.36 46.69 47.28 50.58 39.1</td>
</tr>
<tr>
<td>O</td>
<td>29.56 28.09 30.64 29.41 28.02 30.01 32.76 32.22 30.4 32.25</td>
</tr>
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Table 11  EDS Data Yellow/Brown Textile #4
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<td>Green</td>
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<td>Weight %</td>
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<td></td>
<td></td>
</tr>
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Table 12  EDS Data White/Turquoise Textile #23
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<th>9</th>
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</tr>
<tr>
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<td>1.43</td>
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Table 13 EDS Data Charred Textile #36
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<td>blue</td>
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Table 14 EDS Data Charred Textile #37
APPENDIX D

TEXTILE IMAGES
Plate 4
APPENDIX E

MICROGRAPHS
Plate 15 Colored Rabbit Hair and Milkweed

Legend:
Brightfield: BF; Darkfield: DF, Polarized Light: Pol;
Plate 16 The “Other” Bast and Particulate on Milkweed

Legend:
Brightfield: BF; Darkfield: DF, Polarized Light: Pol;
Plate 17 Fibrillated Fibers, Charred Fibers, Insect and Particulate Deposits

Legend:
Brightfield: BF; Darkfield: DF, Polarized Light: Pol;
Plate 18

Iridescent Colors on Charred Textile

Iridescent Colors on One Fiber
APPENDIX F

SEM-EDS DATA
Comment: Planchette 15, brown, fiber
Comment: Planchette, brown, particulate
Comment: Planchette 15, particulate, internal structure of the rabbit hair visible
Comment: Planchette 16, yellow fiber, large amounts of carbon
Comment: Planchette 16, yellow, particulate spectra
Comment: Planchette 16, yellow, particulate spectra
Comment: Planchette 16, yellow fiber
Comment: Planchette 17, green fiber
Comment: Planchette 17, green fiber, high carbon
Comment: Planchette 17, green fiber
Comment: Planchette 17, green, particulate
Comment: Planchette 18, charred, blue on one end of a fiber
Comment: Planchette 18, charred fiber with orange/red on one end, spectra of particulate matter
Comment: Planchette 18, charred textile, blue (top)
Comment: Planchette 19, blue
Comment: Planchette 19, orange (bottom)
Comment: Planchette 19, fiber, top
Comment: Planchette 19, orange material only
Comment: Planchette 19, charred fiber with no apparent coloration
Comment: Planchette 21, white fiber
Comment: Planchette 21, particulate, amorphous
Comment: Planchette 21, particulate, amorphous
Comment: Planchette 21, particulate, crystal
Comment: Planchette 21, white fiber
Comment: Planchette 21, particulate, amorphous
Comment: Planchette 21, particulate
APPENDIX G

LIST OF MATERIALS USED AND SUPPLIERS
Photography Supplies

Cameras

Canon Rebel 2000 with zoom lens
Canon AE1

Filters

B+W 403 filter special ordered from:

Wratten filters:
B& H Photo, 420 9th Ave, New York, NY 10001.

Film

B& H Photo, 420 9th Ave, New York, NY 10001.

Filter Holder


Flocking Paper

Edmund Industrial Optics®, 101 East Gloucester Pike, Barrington, NJ 08007-1380

Light Sources

Photoflood
B& H Photo, 420 9th Ave, New York, NY 10001.

UV Light Sources

UV Flashlight:
Emissive Energy® Corp., 135 Circuit Drive, N. Kingstown, RI 02852

UV Lights Sources:
SIRCHIE Finger Print Labs., Inc., 100 Hunter Place, Youngsville, NC 27596.
Other Supplies

Rabbit Hair Yarn:
Joseph Galler Inc., Belangor® Col 801 Lot 201021

Rabbit Hair Fiber:
Teena Jennings, 226 West Elm Street, Granville OH 43023-1107

Testfabric:
Testfabrics L-57, Middlesex New Jersey

Double Sided carbon tape
Operating Scissors:
SPI Supplies, Division of STRUCTURE PROBE, Inc., 569 East Gay Street, P.O. Box 656, West Chester, PA 19381-0656