Identification of Dyes on Textiles from RI-100 A Seventeenth Century Narragansett Burial Site

Rebecca T. Johnson-Dibb
University of Rhode Island

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IDENTIFICATION OF DYES ON TEXTILES FROM RI-1000
A SEVENTEENTH CENTURY NARRAGANSETT
BURIAL SITE
BY
REBECCA T. JOHNSON-DIBB

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE
REQUIREMENTS FOR THE DEGREE OF
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TEXTILES, FASHION MERCHANDISING AND DESIGN

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ABSTRACT

Ninety-one woven wool textile specimens from a mid-seventeenth century Narragansett Indian burial site were selected for dye analysis. Transmission spectrophotometry of extracted dye solutions and thin-layer chromatography were the methods of identification for mordant dyes. Non-mordant dyes were reduced in ammonia and sodium hydrosulfite before dilution in butanol for spectrophotometry. Fifty-seven specimens gave positive results in one or both of the tests performed on them. Twenty-eight specimens were positively identified as indigo, twenty-seven as madder. Thirty specimens were undyed and four yielded inconclusive results. Two fragments suggested the presence of the insect dye kermes, one of which may also have been dyed with a hydroxyflavone yellow dye. These results were examined for their implications regarding test methods, historical context, conservation decisions and the applicability of the test methods to small specimens.
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Diane Montenegro provided a curious and funny mind, an acute bibliographic memory and library, an cracked the motivational whip when necessary. Michael Dibb made me laugh at the right moments, gave his time and laboratory and computer expertise, and put up with the mental and physical clutter that goes with graduate school. The author could not have asked for more love and support.
PREFACE

The following research has been presented in the manuscript form as defined in the University of Rhode Island's thesis guidelines. The manuscript has been prepared according to the Guidelines for Authors of the *Journal of American Institute for Conservation*. Following approval, the text will be submitted for publication to this journal.
TABLE OF CONTENTS

THESIS ABSTRACT ii
ACKNOWLEDGEMENTS iii
PREFACE iv
LIST OF TABLES vii
LIST OF FIGURES viii

MANUSCRIPT: IDENTIFICATION OF DYES ON TEXTILES FROM RI-1000, A SEVENTEENTH-CENTURY NARRAGANSETT INDIAN BURIAL SITE 1

1.0 INTRODUCTION 2

2.0 BACKGROUND 3
   2.1 Indian preferences 3
   2.2 Dyes and Dye Trade 6

3.0 PREVIOUS ANALYSIS OF DYES ON ARCHAEOLOGICAL TEXTILES 11

4.0 PROCEDURE 15
   4.1 Preparation and Selection 15
   4.2 Color Classification 17
   4.3 Choice of Analytical Technique 17
   4.4 Identification of Mordant Dyes 18
      4.4.1. Thin Layer Chromatography 19
      4.4.2. Spectrophotometry 20
         4.4.2.1. Spectrophotometer Modifications 20
   4.5 Identification of Non-Mordant Dyes 22

5.0. Results 23

6.0. Discussion 28
   6.1. Analysis of Test Methods 29
   6.2. Results in Context of European-Colonial Trade 31
   6.3. Implications Regarding Contact between Colonists & Narragansetts 34
   6.4. Implications for Conservation 36
LIST OF TABLES

TABLE 1
DYES AND COLORS COMMON IN THE SEVENTEENTH CENTURY 10

TABLE 2
SUMMARY OF RESULTS 23
LIST OF FIGURES

1. Modifications to the spectrophotometer  
2. Spectra of Known Indigo Compared to Unknown Sample 18-47  
3. Thin Layer Chromatogram Showing Separation of Known Madder & Unknown Sample 82-40A  
4. Spectra of Known Madder with & without Magnesium Acetate  
5. Spectra of Unknown 82-40A with & without Magnesium Acetate  
6. Spectra of Sample 36-9A with & without Magnesium Acetate  
7. Longitudinal View showing two fiber colors  
8. Sample 15-42 showing soil particles before analysis  
9. Cross Section of fiber which tested negative for dyes  
10. Cross Section of fiber which tested negative for dye  
11. Specimen showing poor dye penetration of yarns  
12. Specimen showing poor fiber penetration
MANUSCRIPT:

IDENTIFICATION OF DYES ON TEXTILES FROM RI-1000,  
A SEVENTEENTH-CENTURY NARRAGANSETT INDIAN BURIAL SITE
1.0 INTRODUCTION

Interest in the identification of dyes on prehistoric and historic textile has increased within the last twenty-five years. Dye identification has been used to date textiles and place them in cultural context. Identifying dyes on archaeological textiles presents certain challenges. Burial not only causes changes in the color of textiles, but decomposition. Archaeological textiles frequently suffer fragmentation during excavation. Recently, however, technological advances are making dye identification on small specimens more precise.

In the past century interest in Native American culture has resulted in an expanding body of research. Specifically, the material culture of seventeenth century New England Indians has been investigated through the examination of archaeological artifacts. This research combined these two areas of study by analyzing dyes found on extant archaeological textiles from one Native American burial site in southern New England.

Few textiles from the seventeenth century survive, and archaeological textiles are rarely preserved in a temperate climate. Since 1985 the University of Rhode Island Department of Textiles, Fashion Merchandising and Design has been involved in the analysis of the textiles found at several New England Native American burial sites. This researcher analyzed dyes on the extant European woven wool fabrics from a mid-seventeenth century Narragansett Indian burial site in North Kingstown, Rhode Island. The textile fragments from the site, designated Ri-1000, provided a unique opportunity to examine seventeenth century trade textiles. Three laboratory techniques, thin-layer chromatography (TLC) and ultraviolet-visible (UV-VIS) solution spectrophotometry and a specific test for indigotin were employed. Due to the
extremely small textile fragments excavated, modification of the test methods was necessary.

To the naked eye the fragments ranged in color from red-brown to blue and black. Microscopic examination of the textiles revealed that some textiles had mineralized and some of them were originally dyed in shades of red or brown, blue or possibly purple, green and black. Without laboratory testing of the dyestuff, speculation regarding the original colors of the samples could have been misleading. Even with the identification of the dyestuff, the original color and its source could only be guessed as the color produced could vary with mordants, application time or technique and subsequent change in ground. Nonetheless, dyestuff identification provided information regarding Native American consumption of European textiles in the seventeenth century.

The research had four goals. The first was to identify the dyes or dye types on the Rl-1000 textiles using thin-layer chromatography, transmission spectrophotometry and a specific test for indigotin. Evaluation of these analytical techniques of dye identification for application to miniscule specimens was the second goal. Comparison of the findings to the historical record for cultural context was the third objective. Finally, the dyes found were evaluated in terms of conservation decisions regarding the handling and storage of the remaining RI-1000 textiles.

2.0 BACKGROUND

A variety of source material relates to textiles in Colonial America and specifically to Native American use of textiles, both of their own and European manufacture. Writings such as those of trader Roger Williams in Rhode Island and the trade records of Thomas Pynchon in Massachusetts as well as the letters between traders and their factors in Europe suggest that the textile trade with the Indians was specialized and lucrative. In addition, observers
such as Roger Williams provided information regarding popular fabrics and colors.

Documentary sources such as Williams' 1643 *A Key Into the Language of America* indicate that the Narragansetts were an Algonkian tribe who spoke the Narragansett dialect and inhabited most of what is now Rhode Island. By 1620 they were a farming and fishing community of families, seasonally migrating between summers on the coast of Narragansett Bay and inland winter sites.

The trade records of William and John Pynchon, founders of Springfield, Massachusetts, describe trade with New England's Native Americans. These records suggest which fabric types were most popular among their customers, both Native American and Colonial. For example, the Pynchon inventories show that the most common fabrics ordered were "shag" cotton, trading (or trucking) cloth and duffields (Thomas 1985, 182).

Textile and costume historians give definitions of fabric types, while dye historians discuss the availability and popularity of dyestuffs in the seventeenth century. Excavation reports provide burial context, and analyses of grave goods suggest a culture's daily existence. Cultural values or religious practices are sometimes indicated by mortuary practices.

### 2.1 INDIAN PREFERENCES

Records show the Indians of New England were discriminating consumers, selecting fabrics of particular weaves and colors. Even in the sixteenth century, preferences were apparent. In 1524 the explorer Giovanni da Verrazano wrote that "They rated blue and red above all other colors."

(Wroth, 138). Gifts of red coats from the governors of the Massachusetts Bay Colony to Indian leaders were recorded in 1621 and 1638 (Mourt, 60; Winthrop, 271). William Wood wrote in 1635: "If their fancie drive them to
they choose rather a good coarse blanket through which they cannot see, interposing it between the sun and them. . . " (1977, 84-5).

Previous analysis of the RI-1000 textiles indicated that the fabrics duffields (twill weave) and trading or trucking cloth (plain weave) were widely acquired by the Narragansetts (Welters et al. 22-3). The Pynchon trade documents note the yardage of these fabrics, as well as "shag" cotton (a worsted, napped wool fabric), ordered for trade in Massachusetts. Color was frequently specified. Red, blue and white were the most common colors. Further, trading cloth and duffields were preferred in blue, while "shag" cotton was ordered in red almost seventy percent of the time (Thomas 1979, 304). In 1677 Robert Plot described the manufacture of duffields and trucking cloth in England in the following manner:

These duffields . . . otherwise called shags and by the Merchants, trucking cloth, they make in pieces about 30 Yards long and one Yard 3/4 broad, and dye them Red or Blue, which are the Colours that best please the Indians of Virginia and New England . . . (Montgomery 1984, 228).

Regarding color, Superintendent of the Indians of the Massachusetts Bay Colony, Daniel Gookin, wrote in 1674 that trucking cloth was "in that form as our ordinary bed blakets [sic] are made, only it is put into colors as blue, red, purple and some use them white . . ." (152). The preference for red and blue is echoed by Bannister in a 1704 order for wool bays from a London supplier:

. . . after a sort fit for the Indian trade without any Nape with a white stripe through the selvedge . . . but if you see Cause to send any of these they must be all blews . . . Next the blews the red sells best and next the Red the purple (Montgomery 1984, 159).

Striped fabrics were among those found at the Burr's Hill site in Warren, RI (Gibson 1980). Strouds were another fabric traded to the Indians. Several
early eighteenth century records indicate the popularity of red and blue strouts among Native Americans. Red strouts were particularly noted for the quality of color achieved by dyeing with cochineal mordanted with tin (Montgomery 1984, 353). The use of tin as a mordant increased in the seventeenth century (Brunello 1973 202).

Trader Robert Hull repeatedly informed his factors concerning low color intensity, writing that only "good sad colors" or black sold in Boston (Baumgarten 1974, 233-4). Roger Williams noted that the Narragansetts specifically preferred "sad" colors, or those colors "without any whitish hairs" (160).

2.2 DYSES AND DYE TRADE

Although many seventeenth-century Europeans used undyed fabric, numerous textile dyes existed to produce a variety of colors. Mordants, metallic salts applied to the fabrics, were used to enhance the fastness qualities of non-vat dyes and allowed a wider range of colors. Vat dyes did not require mordants and are characterized by their insolubility in water. They require reduction in alkaline sodium hydrosulfite to become soluble and dye fibers. Once on the fibers, vat dyes are returned to their insoluble form by oxidation either in air or with chemical oxidizing agents. The insolubility of vat dyes makes the dyeings fast to wet treatments if applied correctly.

English fabric manufacturers commonly either dyed their goods themselves or sold unfinished goods for coloration elsewhere in England (Kerridge, 163-5). In either case, the dyes in use were derived from natural sources, typically plants and insects. They included logwood bark and tannin with iron and vitriol for black; woad and indigo leaves for blues and purples; madder root and the bark of brazilwood trees for reds, oranges, and browns; weld and fustic plants and quercitron bark for yellows, and dried kermes and
cochineal insects for crimson and scarlet. Woad or indigo was combined with madder to yield red-purples and with weld or fustic to produce greens. Although other dyestuffs also were available, these were the most common in Europe in the seventeenth century (Adrosko, 1971; Wilson 1979, 88).

As a source of blue, indigo, a vat dye, was imported to Europe in large quantities by the early seventeenth century with the establishment of the East India companies (Geijer 1979, 208). Prior to this time the English used woad, a plant which secreted the same dyestuff as indigo, indigotin, but in lower concentrations (Brunello 1973, 145-190); thus the two are chemically indistinguishable. Blues produced with comparable quantities of woad were much lighter than those dyed with indigo. Indigo imports threatened local woad producing industries in Europe, and both the use of indigo and the increasing sales of imported indigo-dyed textiles were outlawed for a period of time in England during the Elizabethan period as well as in France and the Netherlands (Birrell 1973, 385; Adrosko 1968, 45-6; Sandberg 1989, 27-8). Brunello stated that woad and indigo were in competition soon after the discovery of the New World, but he suggests that indigo use did not completely overtake woad until the middle of the eighteenth century (1973, 196; 227).

Logwood, when used with a complex mordant containing iron, copper and aluminum salts, was the first single source of black. Additionally, with a different mordant logwood produced a fugitive dark purple. Derived from the bark of a Mexican tree, it was imported to Europe by the sixteenth century. Although its use was banned in England in 1581 due to its variable fastness, logwood was smuggled into the country for dyeing cloth. Prior to the discovery of logwood, blacks were achieved through numerous overdyeings
of various substances such as nut galls, iron, tannin, vitriol, madder and woad (Brunello 1973, 190; 197; 243).

Madder was the most common source of reds, oranges and browns. Madder was quite inexpensive and, if properly mordanted, reasonably fast to water and light. Its only drawback was that the color range was limited to dull orange reds (Wilson 1979, 91). Between the late fifteenth and the eighteenth centuries the Dutch excelled at madder cultivation and dyeing. They were the primary source for both raw madder and madder-dyed wool (Leggett 1944, 11-13).

Brazilwood was used in Italy as early as the Middle Ages to obtain bright reds. It was imported to Europe from as early as the fourteenth century but produced dyeings that were not fast (Brunello 1973, 130).

Until sometime during the seventeenth century European dyers used kermes, an insect dye exported from the Mediterranean, to achieve pinks and scarlets. Kermes was expensive because the source was the actual insect which had to be plucked at a specific stage of development from the leaves of the plant on which it was feeding. Recent literature has suggested that kermes dyes were made from a variety of kermes insects, although Kermococcus vermilius has been thought to be the source of "true" kermes dye (Cardon 1990, 191-2; Brunello 1973, 199-200).

The alternative to kermes was cochineal, a similar insect found in Central and South America. During the seventeenth century, cochineal superseded kermes, although the date when kermes was no longer used has been debated (Verheeken 1989; 1990; Ziderman 1990). Kerridge says that as late as 1622 madder, kermes and cochineal were all in use in Norwich, England (1985, 168). Spain monopolized the importation of cochineal (which was quite large by the mid-sixteenth century) and traded it to the Flemish and the
French. Cochineal insects produced more dyestuff than kermes, thus fewer insects were required, yet it was less popular in England and Italy than in France and the Netherlands (Brunello 1973, 62; 199-200). Like kermes, cochineal was quite expensive but when properly mordanted, produced a faster, heavier dyeing in bluer shades of red than madder.

Several plants producing yellow dyestuffs grew in England. The most common were fustic and weld (Adrosko 1968, 31-2; 37-9). The colors they produced tended to be light in shade and fugitive to light and water. When overdyed on woad or indigo they produced green, a color for which no single natural source existed (Brunello 1973, 138-9). Another source of yellow, quercitron, derived from the bark of the American black oak, was native to the northern New World and exported to England as early as the sixteenth century (Adrosko 1968, 32-34).

Both the Narragansetts and the Colonists might have colored domestically-made textiles and undyed European textiles with their own dyestuffs. Dyes such as logwood, brazilwood and indigo were available through trade, but most often these dyes were part of a triangle trade between England, the Colonies and the "Spanish Coasts." The Colonists traded goods such as lumber to Central or South America for dyestuffs, sugar and molasses. These were sent on to England for fabric and other trade items before arriving in America (1729 merchant J. Gee, as cited in Baumgarten 1974, 222). Although documents indicate that by 1683 the Colonists traded directly with the West Indies for dyes such as logwood and indigo, these were the same dyes used in Europe (Ibid; Weeden, 1910, 115). Identification of dyes alone does not indicate the location of the dyeing process.
| Dyestuff | Dye Type     | Source                                | Color       |
|----------|--------------|---------------------------------------|-------------|
| Indigo   | Vat Dye      | leaves of Indigofera Tinctoria (India) | lt. to dk   |
|          |              |                                       | blue        |
| Woad     | Vat Dye      | leaves of Isatis Tinctoria L.(Europe)  | lt. to med  |
|          |              |                                       | blue        |
| Madder   | Mordant Dye  | roots of Rubia Tinctoria (Europe)     | orange      |
|          |              |                                       | reds        |
| Kermes   | Mordant Dye  | insect Kermococcus Vermilius (Mediterranean) | blue       |
|          |              |                                       | reds        |
| Cochineal| Mordant Dye  | insect Dactylopius Coccus cacti (S. America) | blue       |
|          |              |                                       | reds        |
| Brazilwood| Mordant Dye  | bark of Brexillium (S. America)       | Br. reds    |
|          |              |                                       | to pinks    |
| Logwood  | Mordant Dye  | bark of Haematoxilon Campechianum (S. America) | dk purple   |
|          |              |                                       | to black    |
| Weld     | Mordant Dye  | Reseda Luteola plant (Northern Europe) | yellow      |
| Fustic   | Mordant Dye  | Rhus Cotinus plant (Northern Europe)   | yellow      |
| Quercitron| Mordant Dye  | Quercus Tinctoria (New World)         | yellow      |
3.0 PREVIOUS ANALYSIS OF DYES ON ARCHAEOLOGICAL TEXTILES

Although interest in the identification of historic dyes has increased among scholars, only a few have addressed the identification of dyes on archaeological textiles. Still fewer have analyzed dyes on North American archaeological textiles. Additionally, synthesis of dye identification with historical information such as chronology, material culture, costume and textile history or religious practices is comparatively rare. One of these areas might be addressed, but wider conclusions seldom are drawn.

Generally, the approach to archaeological textiles has been to use the same test methods as those used on unburied samples. Usually the dye is extracted from the fibers by heating in a solvent mixture or reducing agent solution. The extract is then examined using one or more tests. The most common methods are infrared (IR) or ultraviolet-visible (UV-VIS) spectrophotometry, and high performance liquid (HPLC) or thin-layer chromatography (TLC). Although problems have been noted with these techniques, some adjustments have been made to accommodate archaeological samples. These include abandoning one technique for another, and modifying tests to increase sensitivity for small amounts of extracted dye and make allowance for soiled samples.

An early effort to identify dyes on archaeological fabrics used infrared spectrophotometry. Abrahams and Edelstein (1967) confirmed the presence of alizarin (one of the main components of madder), cochineal (an insect dye), indigo and saffron on wool samples found in the Judean desert and dating to 135 AD. Textiles excavated from desert sites have generally been in better condition and the colors have been brighter than those found in temperate climates. Dye identification on these textiles might have been easier than the identification of dyes on archaeological textiles from wet sites.
Saltzman (1993), Hatchett (1983), Geiss-Mooney and Needles (1981) and Wouters and Rosario-Chirinos (1992) examined textiles from Pre-Columbian sites in Peru. Despite approaching the subject differently and using various methods, all of these researchers agree on the presence of indigo, cochineal and relbunium (a relative of madder). Hatchett, using a modified extraction method and UV-VIS spectrophotometry, focused on certain red-dyed fibers from one mummy found in Paracas, dating 400BC to 400AD. She found relbunium and a single example of cochineal. Geiss-Mooney and Needles used a succession of solvents and UV-VIS to examine late Intermediate textiles and found cochineal, indigo and undyed fibers.

Saltzman (1978; 1993) utilized standard extraction methods and UV-VIS spectrophotometry to test selected fibers from several time periods, primarily the early Paracas period. With a log density scale to minimize concentration effects, he identified shellfish purple, cochineal, relbunium, indigo, a mix of indigo and cochineal and an undetermined yellow dye.

Interestingly, with HPLC Wouters and Rosario-Chirinos found most of the above noted dyes on textiles dating between 300BC and 1532AD and were able to identify Saltzman's unknown yellow as a mixture of several different yellows. Among them were quercitron, luteolin (the dyestuff in the European plant weld) and tannin. Additionally, yellows were found in conjunction with both relbunium and indigo, suggesting that oranges and greens were among the colors of Pre-Columbian textiles.

Although these researchers identified the same dyestuffs to greater or lesser extents, apparently none needed to deal with the limitations of sample size or contamination, and few put their results into a historical context. Wouters and Rosario-Chirinos began to organize their results chronologically.
and found that some dyestuffs were found only on textiles from some limited time periods.

In Europe, many reports of archaeological textile analyses have been published especially since 1969. The primary test methods used are TLC and UV-VIS spectrophotometry.

In an article on the use of thin-layer chromatography and spot tests, Schweppe noted the successful identification of indigo on brown wool fibers from a fifth century BC Celtic grave in Luxembourg (1976, 34). Although his sample weighed less than ten milligrams, he was able to extract indigotin in acetic acid and confirm this result by re-forming an indigotin vat to dye a cotton yarn blue. Unfortunately, the method reported lacks the detail necessary to follow his procedure. Elsewhere (1988), Schweppe described the identification of indigotin through reduction in ammonia and sodium hydrosulfite.

Taylor and Walton have written about their work with Roman (1983), Anglo-Scandinavian (Taylor 1983), Viking-Scandinavian (Walton 1988) and Medieval European (Walton 1984) textiles. They have made comparisons of dye types as well. Lichen purples were the subject of several more articles by Walton and Taylor, the result of discovery at several sites including Roman Vindolanda as well as Viking York. They concluded that lichen purples were in use in Dark Age Europe, despite the research of some historians to the contrary (Taylor & Walton 1983).

Walton (1986) reported the progress of study on Viking Danish textiles dating as early as the pre-Roman Iron Age. She found the dyes comparable to those found in Viking York. Madder and indigotin were found in both places as were combinations of the two. In addition, in Denmark, an unidentifiable yellow dye was found. In a later survey of Viking-age dyes, Walton (1988)
noted that the same yellow was found in Dublin, Ireland, along with madder, lichen purple and indigo. She broke down dye identification results along geographic lines finding variations in the proportions of each of the dyes.

In discussing her results, Walton noted that only about half of the samples tested gave positive results and that those which did not test positive might have been undyed or have been dyed with a dye that did not survive burial. Finally, Walton raised the issue of a representative sample. Given the fragile nature of textiles, Walton questioned if the fabrics found at a site were statistically significant samples of the textiles possessed and used by the inhabitants. Eastwood (1984) raised the same issue in her discussion of the identification of dyes on Egyptian textiles from as early as 1400 BC through 1500 AD. From the identification of dyes and comparison of the number of dyed and undyed samples, Eastwood discussed differences in the range of colors used, as well as the fibers on which they were dyed.

The practical difficulty in identifying yellow dyes has been noted in several articles (e.g. Saltzman 1978; Taylor 1990A; Walton and Taylor 1991). Solution spectrophotometry cannot effectively distinguish yellow dyes on archaeological textiles because, when stained with residual soils, the extract is contaminated, masking absorption at the wavelengths that yellow dyes absorb. Nevertheless, Schweppe (1986) and Walton and Taylor (1991) reported successful identification of yellow dyes with thin-layer chromatography. However, Schweppe was not testing archaeological dyes and despite noting the use of TLC for archaeological textiles, Walton and Taylor did not report specific results.

Pritchard had Walton analyze dyes on medieval Saxon fabrics (1983) as well as sixteenth and seventeenth century textiles (1991) excavated in London. On the Saxon textiles three dyes were found: madder, woad and
orchil (a lichen purple dye). The later textiles produced madder, cochineal, tannin and possibly turmeric and lichen purples. In comparison to fabric quality the more expensive dyes, such as cochineal, were used on the more expensive silk fabrics. Naturally pigmented fibers were not dyed. Additionally, Pritchard cited seventeenth century fashion trends in discussing the finding of cochineal on knitted wool stockings.

Needles, Cassman and Word (1985) are among the few researchers working on textile dyes in North America. They noted problems working with archaeological textiles. In examining Hessian soldier uniform fragments from a Revolutionary War gravesite near Charlottesville, Virginia, they found TLC ineffective in identifying dyes on samples caked with clay even after cleaning. Success was achieved with infrared spectrophotometry. This would seem to underscore the need for a test method which compensates for soils. Taylor's (1990A & B) use of magnesium acetate is effective in intensifying the absorption of certain red dyes but does not help with the identification of yellow dyes in contaminated extracts.

4.0 PROCEDURE

4.1 PREPARATION AND SELECTION

Upon excavation in 1984, professional conservator Dennis Piechota was consulted for treatment of the RI-1000 textiles. On his advice, graduate student Joyce Smith humidified the textiles, treated them with thymol in water to kill bacteria, cleaned them in a five percent ethanolic glycerine solution and consolidated them in ethyl hydroxyethyl cellulose. Before mounting in microclimate storage units for housing at the Rhode Island Historic Preservation Commission, the fragments were characterized according to grave location, color under fluorescent light, fiber content, yarn diameter, yarn twist, weave structure, thread count and finish (Welters 1985, 4-5). Cross-referencing of
these characteristics identified thirty-two different fabrics along with a number of single yarn samples (Welters MS).

For this research, yarn specimens were removed from fragments large enough that characteristics such as weave structure would not be dismantled. In addition, specimens were taken from masses of fibers that had no discernible yarn orientation. No specimens were taken for dye analysis when only fibers or a few yarns remained on matting or other materials. The specimens were categorized by fiber content, quantity of sample and hue.

Two additional specimens were chosen because of recommendations from previous research. A red wool fragment had both mineralized and apparently unmineralized fibers of the same hue. A blue-green fragment exhibited the same phenomenon (Coho 1993). The source of the color was an unanswered question. Several fragments of a single textile exhibited different colors or appeared to include more than one color yarn. Specimens of these textiles were selected for analysis to determine whether the same dye produced different colors with age and burial or if several dyes were present in the same fabric. In all, ninety-one wool specimens were selected for testing.

Fifty-six specimens representing a variety of colors and graves were analyzed using the test methods discussed in the following sections. After experience was gained in the testing procedures, the remaining thirty-five samples were analyzed using either the reduction test for indigotin or spectrophotometry for mordant dyes. The test method for these remaining specimens was selected based on their color under the microscope; that is, blue specimens were tested by reduction in ammonia and sodium hydrosulfite and reds by spectrophotometry.
4.2 COLOR CLASSIFICATION

The RI-1000 fabrics had been categorized according to Munsell Soil Color charts in previous visual analysis under fluorescent light (Welters 1985). In this research color analysis continued with examination under both the stereo microscope and the polarizing light microscope with fibers mounted in deionized water. Specimens were described as blue, green, red, brown or yellow. Based on these color descriptions indigo, woad, madder, kermes, cochineal, weld and old fustic were considered the dyestuffs most likely to be found on the RI-1000 textiles (Brunello 1973; Leggett 1944).

4.3 CHOICE OF ANALYTICAL TECHNIQUES

A wide range of literature was reviewed to select analytical methods. Most notable are Taylor's (1983; 1990A & B; 1991) and Saltzman's (1978; 1992) use and refinements of ultra-violet and visible solution spectrophotometry, plus Hofenk-de Graaff's and Roelof's (1969; 1978) and Schweppes's (1976; 1980; 1988; 1989) use of thin-layer chromatography and spot and lake tests. Wouters (1991) and Whiting (1991) have both used high performance liquid chromatography, and Abrahams and Edelstein (1967) used infrared spectrophotometry.

Based on color, the specimens were categorized as likely to be colored with a vat dye (blues) or a mordant dye (yellows, reds, browns). Two sets of tests were used on each of the first fifty-six specimens, one test on the remaining specimens. The two test methods were chosen to cross reference results. Procedures were selected for their simplicity, cost, speed and suitability for the small size of the samples, generally three milligrams or less.

For mordant dyes solution spectrophotometry and thin-layer chromatography were chosen. Because blues could only have been
achieved with vat dyes, blue and green specimens must have been colored with vat or vat-mordant dye combinations. Therefore blue and green specimens were reduced to solubilize the dye and re-oxidized to identify indigotin. To confirm the indigotin result, the extracted dye was diluted with butanol for spectrophotometry.

Other methods, such as spot or lake tests, were not appropriate as they require much larger samples. Additionally, infrared spectrophotometry and HPLC require expensive equipment not available at the University of Rhode Island's textile laboratory.

4.4 IDENTIFICATION OF MORDANT DYES

Analysis of mordant dyes involved two major steps: extraction of the dye and identification by spectrophotometry and thin-layer chromatography, Extraction of known and unknown mordant dyes was based on procedures outlined by Walton and Taylor (1991). Known dyed wool samples to be used for comparison were obtained by the researcher from the Conservation Analytical Laboratory of the Smithsonian Institution during a course in dye identification with Dr. Helmut Schwepppe in 1990.

A sample was placed in a test tube with one to two milliliters ethanol:10% sulfuric acid (2:1), heated at about 90 degrees Celsius for approximately one hour before cooling to room temperature. The sample was removed, and the extract was shaken with one to two milliliters diethyl ether. After approximately a minute the ether separated the extract into upper and lower layers, with extracted dye in the upper, ether phase. The ether-dye layer was removed with a dropping pipet and evaporated to dryness in a watch glass at room temperature. At this point a few drops of methanol were used to dilute the concentrated dye for TLC.
4.4.1 THIN-LAYER CHROMATOGRAPHY

Thin-layer chromatography was carried out on Baker-flex 6F 5X20 centimeter polyamide-covered plastic plates, cut to approximately five by ten centimeters in length. Prior to dye extraction, separation chambers were prepared with an eluent chosen for the type dye expected to be present. Pre-testing resulted in the following choice of eluents: toluene and acetic acid (9:1) for vegetable reds; butanone-methanol-formic acid (65:35:5) for insect reds (kermes and cochineal); chloroform-methanol-butanone-formic acid (6:2:1:1) for hydroxyflavone yellows (Schweppe 1988, 12). About ten milliliters of the selected eluent was poured in to the 12x4x9 inch separation chamber. The chamber was covered and left undisturbed for one to two hours, allowing the eluent vapor to saturate the chamber.

Using micropipettes, a maximum of one known and three unknown dye specimens dissolved in methanol were spotted on the plates in a line close to one centimeter apart and approximately one centimeter from the bottom and each side. For insect reds one unknown was accompanied by both a kermes and a cochineal specimen.

After chromatograms air dried they were placed in the separation chamber. The chamber was covered, and the chromatogram was allowed to run until the eluent had travelled up the plate surface approximately eight centimeters. Immediately on removal the distance travelled by the eluent was marked with a pencil, and the chromatogram air dried before soaking in 0.5% methanolic uranyl acetate for up to a minute. After unsuccessfully trying ultraviolet fluorescence and potassium acetate, uranyl acetate was found to be the most effective in making faint chromatograms more visible. Finally the chromatogram was examined. Evidence of dyestuffs was noted and compared to knowns.
4.4.2 SPECTROPHOTOMETRY

Prior to testing, several weeks were spent perfecting technique and creating a library of reference spectra from known dyed wool samples. Large and small specimens of each dye extracted from these fibers produced spectra of high and low concentrations of each dye. This range of concentrations showed the variation of spectra that could be expected. The low concentration was chosen to approximate the size of the unknown samples from 1.0 to 3.0 milligrams. Additionally, following Taylor, after the first spectrum, each dye solution was treated with magnesium acetate tetrahydrate and a second spectrum was taken. Magnesium acetate has been shown to cause characteristic intensification of light absorption by certain dyestuffs (Taylor 1983, 155).

Following TLC, the remaining concentrated unknown dye was diluted with approximately four milliliters of methanol, poured into a rectangular glass container called a cuvette and placed into the 2020 Macbeth spectrophotometer. A transmission spectrum was measured and plotted by the spectrophotometer using Macbeth Optimatch software. Additionally, one gram of magnesium acetate was added to the four milliliters of dye solution for a second spectrum. The spectra of the unknowns were compared to those in the reference library, similarly prepared and treated with magnesium acetate, for identification. Results from TLC and spectrophotometry were compared to identification of dyes.

4.4.2.1 MODIFICATIONS TO THE SPECTROPHOTOMETER

Pre-testing showed that although the extraction method was effective, and the addition of magnesium acetate intensified the dyes' absorption of
light, the Macbeth 2020 spectrophotometer (designed as a reflectance instrument) used a cuvette measuring four by three by one centimeters, requiring 10 milliliters of solution when used for transmission measurements. This presented a problem because known specimens the size of the RI-1000 fragments produced ten milliliter extracts that were too low in concentration to produce usable spectra. Modification was necessary.

First small cuvettes, holding approximately four milliliters of solution, were tried. The cuvettes measured one centimeter square and were the same height as the original cuvettes. Because the slot in the spectrophotometer was designed for the larger cuvettes, smaller cuvettes were unsuccessful and yielded variable spectra of the same specimens.

The effective solution was surprisingly simple. A three-sided stand constructed from two-ply board was placed in the bottom of the spectrophotometer’s cuvette slot raising the cuvette high enough to allow four milliliters of solution to intersect the beam of light (fig. 1). However, the empty upper portion of the cuvette, without masking, distorted the spectra. To avoid distortion of the spectra blinders were necessary. Two blinders of the two-ply board were cut to the dimensions of the sides of the cuvettes. Approximately 0.9 centimeter diameter holes were cut in the center of each blinder. The blinders were inserted into either side of the slot in the spectrophotometer with the cuvette in between.

Comparisons of reference spectra made from the extracts of large known specimens without the spectrophotometer modifications and from extracts of small specimens with the stand and blinders showed the modifications did not significantly alter the spectra. The small extract spectra of known dyes were used for comparison with unknowns.
Fig. 1. Modifications to the spectrophotometer.

4.5 IDENTIFICATION OF NON-MORDANT DYESTUFFS

For specimens which appeared blue or green the researcher assumed that the unknown dyestuff was indigotin, either alone or in combination with a mordant dye. Schweppe's (1988, 17) procedure for the reduction of vat dyes was followed. Specimens in test tubes were boiled in approximately two milliliters of concentrated ammonia with about two milligrams sodium hydrosulfite. Specimens with dyes containing indigotin produced a yellow solution of reduced or leuco-indigotin, called a vat. The addition of butanol yielded a blue upper phase within a minute when indigotin (the primary dyestuff in indigo and woad) was present. For confirmation, spectra were
measured. The butanol layer was removed with a dropping pipet, put into a cuvette and diluted with butanol to a quantity of four milliliters for spectrophotometry. Interestingly, lichen dyes and shellfish purple dyes also respond to the vat dye test although they are not actually vat dyes.

After spectrophotometry, fiber specimens were removed from the remaining ammonia, rinsed in water and examined under the stereo microscope for retention of any additional color. Any remaining red or yellow color would have suggested an over-dyeing. None of the specimens exhibited additional color. Had other colors been retained, the mordant dye would have been extracted and tested following the procedure for mordant dye analysis outlined above. Schweppe (1988) has found that the vat dye test does not destroy mordant dyes, allowing the vat dye test to be performed before testing for mordant dyes.

5.0 RESULTS

Ninety-one specimens representing fourteen different graves were analyzed for evidence of dye. Of the ninety-one, fifty-seven gave positive results in one or both of the tests performed on them (see table. 2)

| Tested | Indigotin | Madder | Kermes | Undyed | Inconclusive |
|--------|-----------|--------|--------|--------|--------------|
|        | 91        | 28     | 27     | 2      | 30           | 4             |

Thirty specimens were thought undyed because the spectra of the dye solution extracted by the method considered appropriate due to their original colors did not suggest the presence of dye. When cross-referenced with TLC
and vat test results, the undyed designation was corroborated. Undyed specimens have no dye present to yield TLC results. Four specimens produced inconclusive results in both sets of tests.

Twenty-eight specimens total tested positive for indigotin (fig.2). Of these, two specimens tested positive for indigotin in the vat test, however with the dilution of the butanol to four milliliters, these two extracts were too weak to yield conclusive spectroscopic results. Additionally, after an inconclusive vat test, the spectrum of a third specimen indicated indigotin.

Fig.2. Spectra of known indigo compared to unknown specimen 18-47.

------ Known Indigo.
------- Specimen 18-47.
Twenty-seven specimens tested positive for madder. Of these, thirteen specimens indicated madder by both TLC and spectrophotometry with and without magnesium acetate. The TLC of a known madder and the unknown sample seen in figure 3 is an example. Figure 4 demonstrates the spectra of known madder with and without magnesium acetate. Figure 5 is the spectrum of the unknown dye in figure 3; comparison of figures 4 and 5 confirms the identity of madder suggested by the TLC.

Spectrophotometry alone of twelve specimens indicated the presence of madder. Two specimens tested positive for madder in TLC, but the four milliliter spectrophotometry extracts were too dilute to confirm the TLC results.

Kermes was indicated in the TLC of one specimen, but spectrophotometry was inconclusive. In thin-layer chromatography another sample showed both a hydroxyflavone yellow and an insect red, probably kermes. However, spectrophotometry was unable to confirm these results. No other fragments yielded positive yellow dye results. One sample exhibited neither madder nor insect red results in TLC, but with absorbance maxima at 400 and just over 500 nanometers, its spectrum suggested two dyes (fig.6).

Fig. 3. Thin-layer chromatogram of known madder and unknown specimen 82-40A.
Fig. 4. Spectra of known madder with and without magnesium acetate.
- Madder without magnesium acetate.
- Madder with magnesium acetate.

Fig. 5. Spectra of unknown 82-40A with and without magnesium acetate.
- 82-40A without magnesium acetate.
- 82-40A with magnesium acetate.
At least six samples tested positive for more than one color. In some cases either madder or indigo/woad dyed yarns were combined with undyed yarns. Two specimens had individual yarns dyed different colors. Indigotin and an unidentifiable mordant dye were indicated in one (fig.7), and madder and indigotin were present in separate yarns in another. Two additional fragments may have had two colors present, but the transmission of the solutions were too high to be conclusive. Because of layering of the textiles within the graves or disturbance during excavation some of the fragments
exhibiting two colors originally could have been two separate textiles. However, the identification of two individually colored yarns suggests a woven pattern such as plaid, checks, a border selvedge or stripes such as the blanket fragment found at the Burr's Hill site.

Fig.7. Longitudinal view of specimen 82-49 showing two different color fibers.

Two samples from previous research (Coho 1993) had mineralized sections which appeared under the microscope to be the same color as some unmineralized dyed samples. A blue-green specimen on a brass strip tested positive for indigotin. An extant red specimen similar to red mineralized fibers on a brass fragment in the same grave produced no evidence of dye in spectrophotometry.

6.0 DISCUSSION
6.1 ANALYSIS OF TEST METHODS

This research shows that a reflectance spectrophotometer may be successfully modified to read the transmission of small amounts of solution in low concentrations. Ideally, the analysis would have been performed with a spectrophotometer designed to read small solutions or solutions in low concentration. However, transmission spectrophotometry is a viable tool for identification of dyes on archaeological textiles. The addition of magnesium acetate to dye extracts was found to be useful in obtaining spectra of dyes in low concentrations. As a developer uranyl acetate, widely reported in the literature, was generally adequate although its radioactivity may make its use undesirable.

The selected analytical procedures functioned satisfactorily on approximately seventy percent of the samples. When one test method gave a positive result for dye and another did not, the first analysis may have used all or most of the extracted dye, leaving little for the other test to register. This might have been the result of little dye present in the fibers or incomplete extraction of the dye from the fragment.

Generally, the larger the specimen, the stronger the test results, although this was not always the case. One specimen (fig. 8) weighed almost eight milligrams, yet the extracted dye solution yielded inconclusive results. Weight might be a misleading indicator of the quantity of dyed fibers present. Many specimens were held together by soils and the consolident used in on-site treatment (Welters 1985,3), thus the actual amount of fiber present might have been minimal. Another specimen originally weighed five milligrams yet after testing the remaining fibers weighed only two milligrams due to loss of soils and consolidents.
Inconclusive identification might have been the result of several factors other than the amount of dye present. Yellow dyes are known to be fugitive and difficult to isolate and identify (Walton & Taylor 1991,6; Needles, Cassman & Word 1985) so that the failure rate could be higher than for indigotin or madder. Another explanation is that despite the appearance of color, the samples either were undyed or only partially dyed. Cross-sections of two samples revealed evenly distributed coloration of single fibers in a red-yellow hue with no visible fiber pigmentation (figs. 9 & 10). The lack of positive dye results, when the appearance of color on the specimen suggested dye, indicated soil stains absorbed by the textiles while buried.

Fig. 8. Specimen 15-42 showing soil particles before analysis.
Incomplete dye penetration of the fabric (due to piece-dyeing or unrefined dyeing methods) is another possible reason for inconclusive results.
Microscopic examination prior to dye analysis revealed several specimens with poor dye penetration both of yarns and fibers. The selvedge (fig.11), while predominantly blue, shows several straw colored areas where dye did not penetrate the yarn. In a longitudinal view of a fiber (fig.12), dark blue fades to yellow-green and begins to darken again, indicating that dye did not completely penetrate the length of the fiber.

Although many specimens appeared to be different colors under the microscope, frequently the same dyestuffs were present. Most blue specimens, variously described by researchers as blue, blue-black or blue-green, were, as expected, based on indigotin. Similarly, most specimens described as red, red-brown, orange or dark brown were based on madder. Several factors may have contributed to color differences. Original mordants, amount of dye or fugitive over-dyes would have affected color. Equally important, soils associated with use and burial with grave goods could affect the color of excavated textiles, underscoring the importance of laboratory dye analysis.

6.2 RESULTS IN THE CONTEXT OF EUROPEAN-COLONIAL TRADE

The presence of madder, indigo or woad and probably kermes supports the theory that the woven wool fragments found at RI-1000 were produced and dyed in Europe (Welters MS, 5). Trade records and travel accounts do not indicate either domestic dyeing or dye trade in New England during this period. Additionally, documentary sources do not suggest Native American manufacture and dyeing of woven wool textiles. Madder, indigo, and woad were common dyes throughout Europe in the seventeenth century (Adrosko, 1971). However, it is not possible to determine where the textiles were dyed by the identification of these dyestuffs.
Figs. 9 & 10. Cross-sections of fibers that tested negative for dyes and show no natural pigmentation (82-50; 38-94).
Fig. 11. Poor dye penetration at yarn intersections (specimen 82-50).

Fig. 12. Fiber with uneven dye penetration (specimen 3-20B).
The shift from the use of kermes to cochineal occurred during the seventeenth century. Coupled with the issue of multiple kermes insects, the two kermes results of this research raise some interesting questions. Is the presence of kermes significant? Due to its declining use at this time, kermes must have been hard to find and it was more expensive than madder. This suggests an intentional selection of a kermes-colored fabric. Although depending on the mordant, an insect dye might have been made to produce a color similar to that produced by madder, the cost of using kermes makes this scenario improbable. Alternatively, a kermes-colored textile may have been the only one available at the time of acquisition or burial.

Textiles were valuable and constituted large proportions of the material wealth of European settlers (Thomas 1985, 146; Montgomery 1984). In this context, domestic production was desirable. In fact, efforts were made by the Colonial government to encourage both the raising of sheep and fabric production in the home. However, written records demonstrate that domestic production could not meet the demand for woven fabrics (Baity 1949, 233-4). Merchants such as Roger Williams, the Pynchons and John Hull repeatedly ordered textiles of all sorts from Europe to trade both with Colonists and Native Americans, stressing that the colors and fabric types should be those that sold well both to groups (Welters et al. in press, 26-7).

6.3 IMPLICATIONS REGARDING CONTACT BETWEEN COLONISTS AND THE NARRAGANSETTS

The abundance and quality of grave goods from RI-1000 suggest both that the Narragansetts were wealthy relative to other Native Americans in New England and that the interaction between the Narragansetts and Colonial settlers, or at least traders, was common. The Narragansetts were craftsmen and traders, manufacturing shell beads and wampum, as well as trading furs,
although the most active fur trade occurred farther north (Robinson et al. 1985, 110). Further, researchers suggest that Colonists were reliant on Native American wampum as a currency of exchange between themselves and other Indians (Thomas 1985, 156). The implication is that the Narragansetts were in a position to be selective in their choice of European goods. Documentary sources, such as the records of Robert Plot and Roger Williams and the descriptions written by Gookin, report the Native Americans preference for red and blue fabrics which is supported by the results of this research.

Several interpretations are not mutually exclusive. The use of traditional mortuary practices to reaffirm cultural identity at a time of instability has been related to RI-1000 (Robinson et al. 1985, 122-3). Despite the trend towards traditional burial practices, evidence of European contact is still seen in the textiles found in these graves. Perhaps red and blue textiles were important to the Narragansetts in everyday life and so might have held mortuary significance.

Care must be taken in attributing preferences to the Narragansetts. Although it is not known if these ninety-one specimens constitute a representative sample of Narragansett European textile consumption, the predominance of two particular dyes on the textiles cannot be ignored. Excluding the samples from the fill dirt originally excavated from several graves, madder was found in seven graves, indigotin in five and both dyes were present in four. While madder was commonly available and relatively cheap in the seventeenth century. Cochineal, and, to a lesser extent kermes, also were available but expensive (Hofenk-de Graaff 1969, 83). The finding of two kermes specimens at RI-1000 might reinforce the idea that fabrics colored with madder were preferred. Given that the Narragansetts were selective in trade, a reason for choosing reds and blues must exist. The low cost and
ease of availability of madder, and the depth of shade and color fastness of
colors produced by indigotin make their presence on the RI-1000 textiles
unsurprising.

6.4 IMPLICATIONS FOR CONSERVATION

The extensive post-excavation treatment received by the RI-1000 textiles
does not appear to have affected the identification of the dyes, although the
proportion of conclusive results might have been higher had they not received
consolidation treatment. Post-excavation treatments should be carefully
selected so dye is not lost or chemically altered.

Indigo and madder are two of the most fast natural dyes. Indigotin is not
water soluble in its oxidized form, nor is it vulnerable to photo-degradation
(Bide 1992, 37). Mordanted madder has fair to good fastness to both water
and light (Adrosko 1968, 95-6). In terms of the storage or exhibition of the RI-
1000 textiles, the fibers themselves are probably more of a concern than the
dyes used on them. Fiber degradation is exponential; the more damaged a
fiber, the more vulnerable it is to further degradation (Bresee1986, 41-6). The
RI-1000 textiles have been used, buried, excavated, washed, consolidated
and stored; those fibers which had not mineralized were degraded. The RI-
1000 textiles should continue to be stored or exhibited in conditions
conducive to the preservation of all textiles: constant temperatures and
humidity, with minimal ultraviolet and visible light exposure.

7.0 CONCLUSIONS

Madder and indigotin are the predominant dyes found on the European
woven wool fragments from RI-1000. Additionally, kermes and a
hydroxyflavone yellow dye might have been present. The identification of
these dyestuffs supports both the documentary evidence regarding Native
American color preferences and the probability that these fragments were woven and dyed in Europe.

In terms of analytical techniques, the test methods were satisfactory although, as is often the case, practice in dye extraction and testing was important. Modification of the spectrophotometer allowed the identification of extremely small quantities of dye as well as the confirmation of the other test results. One requirement of both TLC and spectrophotometry is a library of known dyed wool samples from which a set of reference spectra can be prepared with the same techniques as the unknowns. From the same knowns dye may be extracted for use in TLC.
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APPENDIX ONE

LITERATURE REVIEW ON ANALYTICAL METHODS

1.0 SPECTROPHOTOMETRY

Spectrophotometry is an analytical method based on the selective absorption of light. A dyestuff will transmit and absorb light selectively. A spectrophotometric curve of absorbed or transmitted light at different wavelengths can be obtained and compared with absorbance maxima of known dyestuffs. This technique characterizes a dye's behavior when exposed to light.

Several choices regarding the use of spectrophotometry exist. Spectra may be recorded as absorbance or transmission. The wavelengths of maximum absorbance are often a characteristic feature. Transmission spectra have maxima at the wavelengths of least absorbed light. Absorbance measurements are often preferred because absorbance is linear with concentration, potentially yielding quantitative information (Duff & Sinclair 1989, 134).

In this research, quantitative results were not necessary for identification. Such measurements would have reflected the concentration of the dye extracted from the textile as opposed to the amount of dye originally on the textile. Were quantitative information necessary for this research, one of the factors to be considered is the concentration of the dye in the extraction. If the concentration is low, quantitative information may be inaccurate. To compensate a logarithm of absorbance or density may be plotted (Saltzman 1993; 1978; Saltzman & Keay 1967).

Dyes can be identified by analyzing the spectrum of either a textile itself or a solution of dye extracted from the textile. Solution spectrophotometry was most applicable to this project because the specimens, although they have
been cleaned, were small archaeological specimen which retained dirt (Welters 1985, 2-3). Soils often can be separated from dyes in solution, before testing, preventing excessive distortion of a spectrophotometric curve (Taylor 1990A, 1156). Additionally, transmission spectra provide more fine detail (Saltzman, 1993).  

Taylor (1983) enhanced the sensitivity of his method by adding magnesium acetate to the extraction. Magnesium acetate chelates the dye molecules and increases the dye's absorption of light. In transmission spectra the result is a reduction in the percent of transmitted light, so the curve is intensified.

1.1 Methodology

Solution spectrophotometry has been described by several authors for industrial as well as historical research. In all cases, the dye must be extracted from the textile by placing the sample in a solvent and either heating it for a period of time (Taylor 1983; 1986; 1990 A&B; 1991; Saltzman 1967; 1978; 1993) or allowing the solvent and sample to sit at room temperature for a longer period of time (Hatchett 1983). Variations on these methods include using complex solvents (Geiss-Mooney & Needles 1981) or a progression of solvents (Macrae & Smalldon 1979).

The result of extraction is a colored solution the transmission or absorbance of which is measured by the spectrophotometer. It produces a curve of absorbance maxima characteristic of the dye in a particular solvent. The absorbance maxima of the unknown are compared to reference curves of known dyes in the same solvent(s).

1.2 Limitations

Several problems are associated with solution spectrophotometry of dyes on archaeological textiles. The extraction method may be destructive to
the sample, necessitating the use of small amounts of textile. Indeed sometimes only small samples are available. Both circumstances require careful laboratory procedures to extract enough dye to be read by the spectrophotometer. Another limitation is the need for a library of reference samples made from known dyes in a variety of solvents, not always available in the literature. Because dyes produce different curves in different solvents, one curve for each dye may not be sufficient. The production of such reference curves is time consuming.

In addition, a problem has been encountered by some in identifying yellow dyes on archaeological textiles with spectrophotometry. Natural yellow dyes are fugitive, thus producing pale extractions in which contaminants can easily distort the curve. The solution may be so pale that no curve is produced or the resulting curve may be uncharacteristic (Taylor 1990A & B).

The largest drawback of spectrophotometry is in interpreting spectra of combinations of dyestuffs (Feeman 1970). Saltzman (1978) wrote that spectrophotometry can in fact reflect combinations of dyes, if the dyes are soluble in the same solvent. Obviously, if the sample is an unknown, one may not know if more than one dyestuff is present, much less if they are soluble in the same chemical. Clearly, in some cases one would be aware of a mixture: if a sample were green, and only the yellow dyestuff, weld, appeared in the curve, one would know to re-test in different solvents until a blue source was reflected. Additionally, the burial environment might alter colors to the point that although they now appear similar in color, they might contain of a variety of dyes.

Taylor (e.g. 1983; 1990; 1991) developed a system of examination that helps solve the problem of identifying combinations of dyes. His method begins with extraction in pyridine and water. This process will dissolve vat
dyes but will not affect the mordants used with other natural dyes. A spectrum of the pyridine-water extraction is taken. If no dye is extracted, the sample is rinsed and boiled in ten percent sulfuric acid and ethanol after which the solution is shaken with diethyl ether to separate soils from the dyestuff. The ether is evaporated before the dyestuff is dissolved in methanol for a final spectral analysis.

2.0 Thin-Layer Chromatography

Many articles describe the technique of thin-layer chromatography (TLC) primarily for industrial purposes (e.g. Bide & Choi 1992; Brown 1969; Feeman 1970; Macrae & Smalldon 1979; Sweeney 1972). Some articles contain a great deal of practical information on technical refinements (e.g., Rettie & Haynes 1964; Anonymous 1969). More recently, TLC has been used for historical dye identification by several researchers, including Schweppe (1976; 1986; 1989), Taylor (1990A; 1990B; 1991) and Hofenk-de Graaff (1974; 1975; 1978).

Thin-layer chromatography is a tool used to separate organic compounds. Separation occurs when substances in solution (in this case dyestuffs in methanol) are carried across a substrate (cellulose, silica, aluminum or polyamide) through the capillary rise of an eluent (carrier). Throughout the rise of the eluent, solutes are adsorbed from the eluent on to the surface of the substrate at different rates (Rettie & Haynes 1964, 632). Because of its basis in relative rates of adsorption, TLC is useful in identifying mixtures of dyestuffs (Feeman 1970, 84; Rettie & Haynes 1964, 629). In this capacity it is more useful than spectrophotometry.

The mechanism of thin-layer chromatography is a balance of the relative affinities of the three substrates involved: the adsorbent (substrate), the solute (dye) and the solvent (eluent) (Bide & Choi, 133). Affinities are affected by
polarity and molecular geometry in that both influence solubility and the attraction of one substance to another. As the eluent travels across the plate, a succession of equilibria are established between the dye, eluent and adsorbent, resulting in movement of the dye - less where the dye is strongly sorbed and/or weakly interacting with the eluent, and vice versa. A particular dye will thus move a characteristic distance. The technique is simple in terms of equipment and method, and the chemicals and equipment are relatively inexpensive. Practice in preparation and reading results is necessary to produce meaningful results.

2.1 Methodology

Thin-layer chromatography is a simple procedure. Prior to TLC, the dye must be extracted from the textile, concentrated and redissolved in a second solvent from which the dye under test may be suitably spotted onto the plate. Then the extract is spotted onto a glass or plastic plate coated with a substrate (Rettie & Haynes 1964; Brown 1969). Today the plates are usually pre-coated plastic; they are commercially available, and are the most expensive part of TLC equipment.

The dye can be extracted in one of several solvents and usually is concentrated over a steam bath. Methanol is a common diluent. The spotted plate is placed in a separation chamber which has been prepared in advance with the chosen eluent. Approximately ten milliliters of eluent is poured into the 12x4x9 inch closable chamber and allowed to saturate it with vapor for up to two hours. The plate is left in the chamber until the eluent has moved eight to ten centimeters up the plate. This can take between ten minutes and several hours. Once removed from the chamber, the distance travelled by the eluent is marked (Schweppe 1988). Visualization of a colorless chromatogram can be accomplished by exposing the plate to ultra-violet light
when spots are seen as dark areas on a fluorescent background or soaking it in a developer such as methanolic uranyl, magnesium or potassium acetate (Ibid; Bide & Choi, 133).

For identifying historic dyes, several researchers have refined the basic method. Hofenk-de Graaff and Roelofs (1978) developed a system of extraction for both red and yellow dyes. They recommended extracting the dyestuff in boiling 10% hydrochloric acid and then dissolving the concentrated dyestuff in methanol before spotting on silica plates. In the course of their work, they found that for yellow dyes, the most effective eluent was chloroform, ethyl acetate, methylethyl ketone and formic acid in a ratio of 15:5:3:1.

Schweppe (1988) devised a procedure of identification applicable to many types of dyes. TLC was one of the primary methods he employed. Sample preparation begins with boiling the textile in 10% ammonia to cleanse it of contaminants and soils. It is then rinsed in water and methanol, blotted and dried. Next the dye is extracted from the textile first in water, then glacial acetic acid before rinsing in water and boiling in concentrated ammonia and sometimes 10% sulfuric acid. The dyestuff in the most colored of these extracts is concentrated and dissolved in a small amount of methanol for TLC.

Schweppe preferred polyamide plates as the substrate and used a variety of eluents depending upon the extract. For yellow hydroxyflavone dyes, he used chloroform, methanol, butanone and formic acid (6:2:1:1). For red hydroxyanthraquinones two possibilities exist: methanol and formic acid (95:5) or butanone, methanol, and formic acid (65:30:5). He used methanol and uranyl acetate as a visualizer.
Taylor (1990) and Quye (1990) followed Schweppe's basic methods, although they used pyridine and water (1:1) or ethanol and 10% sulfuric acid (2:1) for initial extraction, while Needles and Geiss-Mooney (1981) based their method on Hofenk-de Graaff's technique.

2.2 Limitations

Thin-layer chromatography shares some of the same limitations as spectrophotometry. A set of reference chromatograms, made under the same conditions as the unknowns, is required. Although TLC has been cited as producing quantitative results (Rettie & Haynes 1964), other researchers question its use in this manner (Feeman 1970). However, for the same reasons that quantitative information may not be required from spectrophotometry, the lack of quantitative results from TLC is not a limitation to this research.

A larger issue is the potential for contaminants in the extract. Theoretically, this is not a problem as the method is supposed to separate compounds. However, Needles, Cassman and Word (1985) found that soil adhered to some samples and interfered with TLC. While their method of extraction was not specified, the problem might have been one of laboratory technique that might have been encountered with the RI-100 samples. Still, Taylor (1990) found extraction and TLC sufficient to separate soil from dye and identify yellow dyes unidentifiable by spectrophotometry. Careful extraction and separation of the extract with ethyl acetate or ether might eliminate contamination problems.
1.0 Molecular and Physical Structure of Wool

The molecular and physical structure of wool and its chemical composition directly affect the ability of wool to be dyed. On the molecular level, wool is a protein called keratin, made up of seventeen to eighteen different amino acids held together in a helical formation by hydrogen bonds. Together with these bonds, two kinds of cross-links between helices provide wool with many of its physical characteristics.

The sulfur atoms in the amino acid cystine form covalent cross-links called cystine linkages. The cystine links are vulnerable to damage by alkalis. Ionic links occur between amine and carboxyl groups to form salt bridges. Together the cystine and salt linkages make wool extremely resilient. At pHs outside the pH4-5 iso-ionic region an excess of positively and negatively charged functional groups are available to react with mordants or dyes (Kadolph et. al 1993, 58-59).

Based on its molecular structure, wool should be a simple fiber to dye. Due to its physical structure, this is not the case. Each wool fiber has essentially four parts. In the center is a hollow or semi-hollow core called the medulla. Cook says that generally the smaller the medulla the finer the wool and that in modern Merino wool the medulla may be invisible or absent (101-2). Surrounding the medulla is the cortex of the fiber, the area of keratin deposition. It is made up of two regions (called ortho or para) of fibrils which either twist around each other or form a sheath and core. Encasing the cortex is the cuticle, and the epicuticle, the endocuticle and the exocuticle which
make up the scales. Scales are responsible for the felting abilities of wool, and together with the epicuticle membrane repel moisture (Cook 1984, 98-102).

2.0 Wool Dyeing

For dyeing to occur, the cuticle must be penetrated, and dye sites must be available. Heat and moisture will loosen the cuticle to allow the dyes to penetrate, thus most wool dyeing involves boiling in water. The links are pH dependent. At pHs below 4-5, dye penetration and exhaustion are increased by the provision of an excess of positively charged sites. Thus acids often are added to wool-dyeing solutions.

2.1 Mordants

Prior to Perkin's discovery of mauvines in 1856, all dyes were derived from natural mineral, animal or vegetable sources. Applied alone most of these dyes were not very fast to light and water. Thus mordants were applied to aid fastness properties. The first use of mordants has not yet been documented, however Middle Kingdom Egyptians (2000-1500 B.C.) are known to have used them (Adrosko 1968,4).

Mordants for wool are metal ions, commonly aluminum, chromium, iron, tin, or copper which function in several ways to yield generally fast dyeing. The mordant is applied to the wool fibers in the form of metallic salts dissolved in a heated bath. During the dyeing the metal cations react with the wool (Knecht & Fothergill 1924, 189). Later, in a warm acidic dyebath, the dye molecule reacts with the mordanting cation and is precipitated on to the fiber in the form of insoluble lakes (Gohl & Vilensky 1990, 82; 148). The resulting metal-dye complexes are chemically more stable due to a sharing of electrons (Aspland 1993, 56) and have increased dye-fiber interaction. Greater interaction makes acid dyes more resistant to alkaline substances.
such as laundry detergents (Gohl & Vilensky 1990, 83). Additionally, several of the mordanting metals react with more than one dye molecule producing dye-metal-dye complexes too large to exit the polymeric structure (Smith & Black 1982, 342).
APPENDIX THREE
HISTORICAL BACKGROUND

1.0 ENGLAND AND NEW ENGLAND

The political situation between England and America was tense in the seventeenth century. Elizabeth I died in 1603 and was succeeded by James I. His son, Charles I, ruled until his death in 1648. Both James I and Charles I were lavish spenders and believers in the absolute power of the monarchy. Despite England's rising economic fortunes, parliament distrusted royal reports of fiscal need, setting the stage for conflict (Palmer & Colton 1961, 144ff). At the same time, Calvinist religious theory, while being suppressed by the Anglican church, was gaining popularity among the middle class. British emigrants, fleeing what they felt was a repressive monarchy in Great Britain, founded the Plymouth Colony in 1620. They were the English country-people known as Separatists for their Calvin-inspired religious beliefs. Prior to coming to the New World, some had lived in Holland for several years (Stratton 1986, 19). Many were so poor that they could not afford the passage on the Mayflower and consequently were mortgaged to London merchants for seven years after their arrival (Warwick et al. 1965, 95). Additional settlers arrived in New England, mainly from Britain, sporadically throughout the century.

Rhode Island as a collection of communities was established by outcasts from Massachusetts. Roger Williams, founder of Providence, was banished from Puritan Massachusetts Bay Colony in 1635. He was considered a heretic for advocating separation of church and state. On the advice of his friend, Massachusetts Governor John Winthrop, Williams headed south and lived among the Narragansetts before purchasing land from them, in 1636-37,
and calling it Providence. Eventually he started a trading post not far from the RI-1000 site in Wickford, patronized by both the Narragansetts and fellow settlers in Rhode Island. Anne Hutchison, similarly convicted by the court of Massachusetts for sedition and contempt, was excommunicated in 1638. With her family, she also went south, founding the Rhode Island community of Portsmouth (Weeden 1890).

1.1 Fabric Production and Trade

After the arrival in the Colonies, the hardships of survival left little time for Colonists to rear sheep, and produce cloth (Leggett 1947, 234). Thus early settlers were largely dependant on Europe, specifically England and The Netherlands, for goods they could not produce themselves (Baity 1949; Walton 1925). To the British, the colonies represented a captive market, and they dumped out-of-fashion goods on the colonists. Additionally, while not dependent on American trade, the British monopolized American trade, imposing tariffs and trade regulations for their own benefit (Baumgarten 1974, 225). Ironically, as early as 1640 the British trade monopoly stimulated some New England sheep rearing and wool fabric production (Leggett 1947, 237). A Massachusetts Bay survey estimated the sheep population in that colony at over 1000 by 1643. The Colonists' hope was to make their own wool fabric. Toward this end the first wool fulling factory was established in 1643 in Rowley, Massachusetts. Massachusetts Bay settlers traded with Spanish traders for raw wool and with the Dutch settlers of Manhattan for the Texel breed of sheep to enhance their breeding stock (Ibid, 238).

By 1675, New Englanders were trading their surplus raw wool to Spain, Portugal and France for linens, silk fabrics, brocades, wine and spices. This prompted even stricter trade regulations from England. In Rhode Island, both weaving and trade were of primary importance with regard to textiles.
Although later than the RI-1000 burials, records show that by 1683, not only were most households engaged in cloth production, they also traded for "... linsey-woolseys and other coarse cloths from Massachusetts" (Weeden, 1910, 115).

Nevertheless, although Colonial Americans produced woven textiles, merchants' records from the time show that the quantities of domestically produced textiles did not satisfy the need (Baumgarten 1974, 220; Welters et al. in press, 1-3). Indeed, Colonists purchased European textiles in large quantities for clothing, home use and probably trade with Native Americans. The letters of the Merchant John Hull, although not differentiating between Native American and Colonial preference, repeatedly request a wide variety of fabric. Other merchants ordered textiles specifically for their Indian patrons. Roger Williams noted a Narragansett preference for "sad colors without any whitish haires" (Welters 1985, 26) which is echoed in the descriptions of Gookin (1970, 152) and Bannister (Montgomery 1984, 228).

No evidence indicates that the Narragansets wove fabrics from animal fibers. Instead, they plaited and twined grasses and rushes into mats and cordage. Because of depletion of the fur-bearing animals in the region and exposure to European traders, they began to appreciate the wearing qualities of woven textiles. Eventually, they acquired woven wool textiles from traders such as Roger Williams who procured them from English or Dutch merchants (Turnbaugh, 1993, 133).
APPENDIX FOUR
THE RI-1000 TEXTILES

During the summer of 1982 and 1983 the burials of fifty-six individual Narragansett Indians were excavated in Rhode Island. The site, called RI-1000, located in North Kingstown, is one of only four known Native American cemeteries of the historic period in southern New England. Both skeletal remains and grave goods were unearthed from the site, and when analyzed, dated the site to 1650-1670 (Turnbaugh, 1984). Found in the graves were goods such as buttons, pipes, brass kettles, spoons and glass beads as well as seventy-six fragments of nine different types of European woven wool textiles and cotton fabrics (Welters MS, 6-10).

Other Native American burial sites have yielded woven wool fabrics as well as some luxury textiles of European manufacture (Gibson 1980). Until recently the woven wool fabrics in Native American cemeteries have been overlooked (Welters et al. in press). Upon excavation, the RI-1000 textiles were cleaned, consolidated, mounted and stored at the Rhode Island Historic Preservation Commission in Providence.

Subsequently Welters (1985; MS) characterized the textiles according to yarn size, yarn twist, fabric weave, thread count and finish. The specimens were assigned color notations according to Munsell Color Charts. Coho (1993) examined those textiles which had mineralized, including some descriptions of color. However, the presence of dyes were not confirmed. Preliminary examination of the RI-1000 textiles showed mainly heavy, coarse wool fabrics. Most appeared stained by grave conditions, but shades of red, blue and brown were evident in others. Some specimens which appeared blue showed yellow under the microscope, suggesting that green fabrics
might also have been acquired. The natural color of the wool might have been yellow enough to produce greens when dyed with indigotin.
# APPENDIX FIVE
## TABLE OF DYES FOUND AT RI-1000

| Sample Number | Fabric Type | Munsell Color | Color Tungsten | TLC/Vat Results | Spectro-Results |
|---------------|-------------|---------------|----------------|-----------------|----------------|
| 3-3A          | Coarse      | 10YR4/4       | Yellow/Brown   | ?               | Undyed         |
| 3-3B          | Plain       | 5Y2.5/2       | Blue           | Indigotin       | Indigotin      |
| 3-3C          | Weave       |               | Blue           | Indigotin       | Indigotin      |
| 3-18B         | ?           | 7.5YR3/4      | Blue           | Indigotin?      | Indigotin?     |
| 3-20A         | Coarse 8x6 plain weave | 10YR3/3-2/2 | Undyed         | ?               | Undyed         |
| 3-20B         | Weave       | Blue-black    | Indigotin      | Indigotin       | Indigotin      |
| 3-21          | Coarse 6x5 plain | 5YR3/2       | Green          | Indigotin       | Indigotin      |
| 9-5           | Full wool?  | 5YR4/4        | Reddish        | ?               | Undyed         |
| 11-8A         | ?           | 10YR3/3/4     | Undyed         | ?               | Undyed         |
| 11-8B         | ?           | Greenish      | ?              | Undyed          |
| 11-8C         | ?           | Red-brown     | Kermes &       | ?               | Undyed         |
| 11-9A         | Coarse      | 10YR3/3-4     | Undyed         | ?               | Undyed         |
| 11-9B         | weave?      | Red-brown     | Madder?        | ?               | Undyed         |
| 11-9C         |            |               | ?              | Undyed          |
| 11-10A        | Nap 7x7     | 7.5YR4/2      | Reddish        | ?               | Undyed         |
| 11-10B        | Plain Weave | Yellowish     | ?              | Undyed          |
| 15-35         | Worsted?    | 10YR3/3       | Red & Greenish | Kermes?         | ?              |
| 15-42         | Coarse      | 7.5YR3/2      | Reddish        | ?               | Undyed         |
| 15-44         | Napped      | 10YR3/3       | Red-brown      | ?               | Undyed         |
| 17-23         | Coarse 6x5 plain weave | 5Y2.5/1     | Blue           | Indigotin       | Indigotin      |
| 17-25         | Napped 5x3 plain weave | 5Y2.5/1     | Blue           | Indigotin       | Indigotin      |
| Sample Number | Fabric Type | Munsell Color | Color Tungsten | TLC/Vat Results | Spectro-Results |
|---------------|-------------|---------------|----------------|-----------------|----------------|
| 17-26W 7.5YR3/2 Blue | Indigotin | Indigotin | Undyed | |
| 17-26F Napped 7.5YR3/2 Orange | Indigotin | ? | Undyed | |
| 17-27 Coarse 5Y2.5/1 Blue | Indigotin | Indigotin | Indigotin | |
| 18-44A 7x7 5Y3/1 Blue | Indigotin | Indigotin | Indigotin? | |
| 18-44B Plain 5Y3/1 Blue | Indigotin | Indigotin | Indigotin? | |
| 18-44C 7x7 10YR3/2 Undyed | Indigotin | ? | ? | |
| 18-47 7X5 Plain 5Y5/1 Blue | Indigotin | Indigotin | Indigotin | |
| 19-16 Heavy nap 2.5YR 2.5/2 Red-brown w/soil | Madder | Madder | Madder? | |
| 21-6A 8x7 Plain Weave 5YR6/3 Reddish | Madder | Indigotin | Indigotin | |
| 21-6B Weave | Bluish | Indigotin | Indigotin | |
| 21-7 8x8 Plain Weave 5YR3/2 Reddish | Madder | Indigotin | Indigotin | |
| 21-8A 5x5 nap Plain Weave 10YR4/3 Undyed | Indigotin | Undyed | Undyed | |
| 21-8B Weave | Purpley | Indigotin | Undyed | |
| 32-5 8x8 Plain Weave 7.5YR3/4 Reddish | Indigotin | Undyed | Undyed | |
| 36-9A 8x8 Plain Weave 7.5YR3/4 Red-brown | Indigotin | Madder | Madder | |
| 36-9B Weave | Blueish | Indigotin | Madder | |
| 36-10A ? 5YR3/2 Blueish | Indigotin | Madder | Madder | |
| 82-40A 7x7 Plain Weave 5YR2.5/2 Red | Madder | Madder | Madder | |
| 82-40B Weave 5YR4/3 Reddish | Madder | Madder | Madder | |
| 82-42 5x5 Plain Weave 5Y2.5/2 Blue-green | Indigotin | Indigotin | Indigotin | |
| 82-47 Coarse 5Y4/1 Blue | Indigotin | Indigotin | Indigotin | |
| 82-49A 7x6 Plain Weave 2.5YR2.5/2 Reddish | Indigotin | Indigotin | Indigotin | |
| 82-49B Weave | Blue | Indigotin | Indigotin | |
| 82-51 7x7 Nap Plain weave 10R3/2 Red | Madder | Madder | Madder | |
| Sample Number | Fabric Type | Munsell Color | Color Tungsten | TLC/Vat Results | Spectro-Results |
|--------------|-------------|---------------|----------------|-----------------|----------------|
| 82-52        | 6x6 Plain Weave | 10R2.5/1       | Red            | Madde           | Madder         |
| 82-54        | Plain Weave    | 2.5YR3/2       | Red-brown      | Madder          | Madder         |
| 82-60A       | Coarse Wool    | 10R3/3         | Red            | Madder          | Madder         |
| 82-60B       | Coarse Wool    | 10R3/3         | Red            | Madder          | Madder         |
| 82-62        | Napped         | 10R3/3         | Red            | Madder          | Madder         |
| 82-63        | Coarse Heavy Nap | 2.5YR3/4     | Red            | Madder          | Madder         |
| 82-64        | Coarse Heavy Nap | 10R3/3       | Red            | Madder          | Madder         |
| 82-66A       | 5x5 Napped Plain Weave | 10R3/4 | Red            | ?               | ?              |
| 82-144B      | 6x6 Plain Weave | 2.5YR2.5/2      | Red bumps      | ?               | Madder         |
| 82-145       | 5x5 Plain Weave | 10YR3/2-7.5YR 4/4 | Blue          | Indigotin       | Indigotin      |
| 82-151       | 6x6 Napped Plain Weave | 2.5YR3/4   | Red            | Madder          | ?              |
| Sample Number | Fabric Type | Munsell Color | Color Tungsten | Vat Results | Spectro-Results |
|---------------|-------------|---------------|----------------|-------------|----------------|
| 11-12A        | Coarse      | 5YR3/1        | Blue           | Indigotin   | Undyed         |
| 11-12B        | 7x7 Plain   | 5YR2.5/12     | Undyed?        | Brown       | Madder         |
| 11-12C        | Weave       | 7.5YR5/8      | Ochre Deposits | Undyed w/Ochre |
| 15-46         | Coarse      | 10YR3/2       | Bluish         | Indigotin   |                |
| 32-4          | Fine        | 5YR3/4        | Orange         | Madder      |                |
| 32-7          | Looped plain| 10YR5/4       | Undyed?        | Undyed      |                |
| 32-8          | Weave       | 10YR5/4       | Undyed         | Undyed      |                |
| 33-3          | Fine 10x10  | 2.5YR2/4      | Greenish       | Undyed      |                |
| 33-4          | Same as 33-3| 5YR3/4        | Brown          | Undyed      |                |
| 38-94         | Napped twill?| 2.5YR3/4      | Brown          | Undyed      |                |
| 50-5          | Fine 12x12  | 5YR3/6-10YR3/2| Reddish       | Undyed      |                |
| 50-6          | Fine 2x1    | 7.5YR3/4      | Reddish        | Madder?     |                |
| 82-41         | Plain 8x8   | 10YR4/3       | Reddish?       | Madder      |                |
| 82-43         | Coarse 4x4  | 5Y2.5/2       | Blue           | Indigotin   |                |
| 82-44A        | Napped      | 10YR3/2       | Undyed         | Madder      |                |
| 82-44C        | 7x7 Plain   | 10YR3/2       | Blackish       | Indigotin   |                |
| 82-45         | See 82-44   | 10YR4/3       | Undyed         | Madder      |                |
| 82-46         | Napped      | 7.5YR4/6      | Orange         | Madder      |                |
| Sample Number | Fabric Type | Munsell Color | Color Tungsten | Vat Results | Spectro-Results |
|---------------|-------------|---------------|----------------|-------------|----------------|
| 82-50         | Coarse 7x5 Selvedge | 5Y2.5/1 | Blue | Indigotin | |
| 82-53         | Coarse 5YR3/2 | Brown | Undyed | |
| 82-56         | Coarse 10YR3/3 | Blue | Indigotin | |
| 82-57         | Napped 2.5YR5/4 | Reddish | Madder | |
| 82-58         | See 82-57 | |
| 82-66B        | Napped 7x7 Plain Weave | 7.5YR3/2 | Brown | Madder? | |
| 82-74         | Coarse 2.5YR3/2 | Red-brown | Madder | |
| 82-79B        | Coarse 10YR3/3 | Tan | Indigotin? | |
| 82-79E        | 8x8 Plain Weave | 10YR3/3 | Brown | Undyed | |
| 82-79H        | | 10YR4/3 | Tan | Indigotin | |
| 82-144A       | Coarse 6x6 Plain Weave | 2.5YR2.5/2 | Red | Madder? | |
| 82-146        | Napped 5x5 Plain Weave | 10YR2/2-10YR4/3 | Brown | Indigotin | |
| 82-149        | Coarse 8x7 Plain Weave | 5YR2.5/2 | Tan-brown | Indigotin | |
| 82-150        | Coarse 8x5 Plain Weave | 10YR4/4 | Undyed | Undyed | |

? = no dye detected
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