Research Design: A Pilot Study for Identifying Raw Material Source Areas for Prehistoric Artifacts from Fort Bragg, North Carolina

(third draft of November 4, 2001)

Introduction

As outlined in the scope of work, the goal of this investigation is to characterize the compositional variation in the metavolcanic rocks and the ceramic clays that would have been used by the prehistoric inhabitants of the Fort Bragg vicinity. This goal will be achieved by examining the chemical and mineralogical composition of 50 rock samples from various quarries in central North Carolina, as well as 50 sherd samples from Fort Bragg and neighboring regions. A study of this nature must be viewed as a pilot study, one that will give a preliminary indication of the range of variation in these materials, and a preliminary sense of which analytical techniques work best in discriminating the potential sources. Specific questions regarding *which* sources were actually used by Fort Bragg's ancient inhabitants must await a future study.

In the paragraphs that follow, we will first outline how the lithic and ceramic samples will be chosen. Then we will briefly review each of the analytical techniques that will be used. Vitas for the principal project researchers are provided as attachments to this document.

Sampling and Analytical Strategy

The sampling will be coordinated by Vincas Steponaitis and R. P. Stephen Davis at the Research Laboratories of Archaeology (RLA), UNC-Chapel Hill. All ceramic samples and about half the rock samples will be drawn from existing archaeological collections, mostly at Fort Bragg and the RLA. The remaining rock samples will be collected in the field during quarry visits coordinated by Chris Moore at Fort Bragg.

Ceramics

The ceramic sample will consist of archaeological sherds drawn from three different drainages. One drainage (the Cape Fear) will be sampled more intensively, and two drainages (the Lumber and Yadkin) less intensively. The former sample will give a sense of the range of variation that may exist within a single drainage; the latter samples will allow us to assess the differences that may exist between drainages. Ten sherds are considered a minimal sample to characterize a single drainage or vicinity. Note that the Cape Fear sample will also be spread across three periods (Early Woodland, Middle Woodland, and Late Woodland), in order to assess how materials may have changed through time. A list by category of the proposed samples is presented in Table 1.

Each sherd in the sample will first be photographed and described. Then each sherd will be sawed into three pieces. One piece will be retained (in the original collection) as a reference, a

second piece will be thin sectioned and subjected to petrographic analysis, and the third piece will be powdered and subjected to instrumental neutron activation analysis (INAA).

Lithics

The lithic sample will consist of rocks collected at three different *quarry groups,* each of which can be subdivided into *quarry clusters*. As with the ceramics, one group (Uwharrie) will be sampled more intensively in order to assess intra-group variability. Two other groups (U.S. 501 and Chatham) will be sampled less intensively so as to allow for inter-group comparisons. In this case, 12 samples will be considered the minimal number to characterize a group, and 5 the minimal number to characterize a quarry cluster. The geographical distribution of samples is presented in Table 2. The locations of the main quarry groups and clusters are illustrated in Figures 1 and 2.

Each rock sample will be photographed and described. It will be then sawed into three pieces. One piece will be retained (in the original collection) as a reference, a second piece will be thin sectioned and subjected to petrographic analysis, and a third piece will be powdered. The powder will then be split and subjected to two kinds of analysis: instrumental neutron activation analysis and Nd-isotope analysis.

Analytical Methods

Once the samples are collected, a number of analytical methods will be employed to characterize them, as described below. Prior to these studies, the sherd samples will be photographed and described by Joe Herbert at Fort Bragg. The rock samples will be photographed at the RLA as necessary.

Neodymium-Isotope Analysis

This analysis measures the ratio of two isotopes of the element neodymium: ^{143}Nd and ^{144}Nd. In igneous rocks, this ratio depends on the source of the magma from which the rock was formed and the rock's geological age. The hope is that different quarry groups or quarry clusters will exhibit distinctive ratios that will allow them to be chemically discriminated. The Nd-isotope study will be carried out on rock samples by Brent Miller at the University of North Carolina at Chapel Hill.

For neodymium (Nd) isotopic analyses, samples are crushed and pulverized to a fine powder in an Aluminum-oxide shatter box. Approximately 200 mg of sample powder is dissolved with an HF/HNO_3 mixture in pre-cleaned teflon high pressure dissolution vessels, and then a mixed ^{147}Sm-^{150}Nd tracer solution is added. Complete dissolution is achieved after 7 days at approximately 180˚C. Conversion from fluoride to chloride is achieved in the same dissolution vessel using HCl. Separation of bulk REE group follows standard cation exchange procedures.

Nd and Sm are separated using 2-methyllactic acid on cation exchange resin. Analytical procedural contamination is less than 200 pg for Sm and Nd, which is negligible considering the Sm and Nd concentrations of analyzed samples. Isotopic analyses are performed on an eight-collector VG Sector 54 magnetic sector, thermal ionization mass spectrometer in dynamic-multicollector mode. Typical ^{144}Nd beam intensities are $2.5E^{-12}$ to $3E^{-11}$ volts relative to a 10^{-11} ohm resistor. Replicate analyses of the Ames and La Jolla Nd standards yield $^{143}Nd/^{144}Nd$ of 0.512143 ± 0.000009 and 0.511850 ± 0.000008 respectively. Neodymium isotopic compositions are normalized to $^{146}Nd/^{144}Nd = 0.7219$ using an exponential fractionation law. Internal run precision for Nd is better than ±0.000005, absolute.

Instrumental Neutron Activation Analysis

Instrumental Neutron Activation Analysis (INAA) is a highly precise and sensitive technique for measuring the concentrations of elements. It involves irradiating the sample with thermal neutrons (usually from a nuclear reactor) and measuring the energies of the gamma rays that are emitted. This technique is widely used in archaeological sourcing studies. For the present study, this analysis will be carried out at the Missouri University Research Reactor (MURR) by Michael Glascock (lithics) and Hector Neff (ceramics).

The powder samples are oven-dried at 100 degrees C for 24 hours, then placed in a vacuum dessicator to cool. Portions of approximately 200 mg are weighed and placed in small polyvials used for short irradiations. After completion of the short irradiation, the samples were transferred to high-purity quartz vials used for long irradiations and reweighed. Along with the unknown samples, reference standards of SRM-1633a (coal fly ash) and SRM-688 (basalt rock) are similarly prepared, as are quality control samples (i.e., standards treated as unknowns) of SRM-278 (obsidian rock).

Neutron activation analysis of pottery at MURR consists of two irradiations and a total of three gamma counts. A short irradiation and count is carried out through the pneumatic tube irradiation system. Samples are sequentially irradiated, two at a time, for five seconds at a neutron flux of 8×10^{13} n/cm^2/s. Following the irradiation, each sample is allowed to decay for 25 minutes before initiating the count on a high-resolution germanium detector. The 720-second count yields gamma spectra containing peaks for the short-lived elements Al, Ba, Ca, Dy, K, Mn, Na, Ti, and V. Following a two-week decay, all specimens are transferred to high-purity quartz vials, as mentioned above. The entire batch of samples is then subjected to a 24-hour irradiation at a neutron flux of 5×10^{13} n/cm^2/s. After the long irradiation, samples decay for seven days, then are counted for 2000 seconds (the "middle count") on a high-resolution germanium detector coupled to an automatic sample changer. The middle count yields determinations of seven medium halflife elements, namely As, La, Lu, Nd, Sm, U, and Yb. After an additional three- or four-week decay, a final count of 10,000 seconds is carried out on each sample. The latter measurement yields the following 17 long halflife elements: Ce, Co, Cr, Cs, Eu, Fe, Hf, Ni, Rb, Sb, Sc, Sr, Ta, Tb, Th, Zn and Zr. Thus, 33 elements are measured in all.

Petrographic Analysis (Ceramics)

Petrographic analysis entails examining thin sections under a polarizing microscope. It is a standard method of identifying minerals (and other substances) in archaeological pottery. This analysis will be carried out by Michael Smith at the University of North Carolina at Wilmington.

Standard (27 x 46 mm) petrographic thin-sections are prepared in a manner so that both the inner and outer surfaces could be examined. Because of the friable nature of some sherds, epoxy impregnation can be used to bind the sample. The thin sections are examined using some of the techniques discussed by J. B. Stoltman (1989, "A Quantitative Approach to the Petrographic Analysis of Ceramic Thin Sections," *American Antiquity*, vol. 54, pp.147-160). Grain size values are very fine (< 0.0625 mm), fine (0.0625 - 0.25 mm), medium (0.25 - 0.49 mm), coarse (0.50 - 1.0 mm) and very coarse (> 1.0 mm). Color identification (hand sample) is based upon the Munsell color chips (GSA, 1991) and colors are observed under fluorescent lamps and described from a dry surface.

The component categories used are paste, rock fragments (three possible types: igneous, sedimentary and carbonate), mineral grains (quartz, plagioclase feldspar, potassium feldspar, mica, mafic minerals, opaque minerals, and unknown), fossil fragments, and grog. The paste is evaluated as either aplastic component at the 0.1 mm size and smaller or amorphous glass. Mineralogy or textural features are used to identify the rock and fossil fragments. The mineral grains, such as quartz, are separated using criteria such as monocrystalline vs. polycrystalline texture, grain size, and degree of angularity and rounding of corners. Mica is identified as either muscovite or biotite based upon optical properties. In this study the feldspar minerals will be identified based upon the presence or absence of diagnostic twinning. If there was no twinning it will be described as feldspar. Plagioclase feldspar is identified by characteristic albite polysynthetic twinning and alteration mineral assemblage while lack of polysynthetic albite twinning and/or presence of Carlsbad twinning identified potassium feldspar (K-spar). Sometimes separation of the feldspars can be made using 2V determinations. For the most part, 2V determinations of the feldspars are affected by the firing process and may not prove to be definitive.

Although the percentage of void spaces is sometimes used as a characteristic, it is often very difficult to use with sherds. A problem associated with any thin-section (or macroscale) investigation is the difference thicknesses of the sherds being analyzed, which may also result in differences in percentage of void space. Nevertheless, a strictly qualitative evaluation of void spaces will be completed to allow the investigator to compare paste versus temper distribution, as well as the potential for shell material (or other components) that may have been lost through firing or dissolution.

Petrographic Analysis (Lithics)

Petrographic analysis can also be used to identify the mineralogical composition of rock samples. This analysis will be carried out by Edward F. Stoddard at North Carolina State

University.

From each sample selected, a slice approximately 1.5 cm thick will be produced using a lapidary saw. From the slice, a rectangular billet will be cut, no larger than 27 x 46 mm. Billets will be submitted to a commercial lab for production of petrographic thin sections. Thin sections will be permanently mounted on a glass slide and have a coverglass. Remaining rock material, including the used billets, will be saved and labelled. Petrographic analyses will consist of:

1. A brief macroscopic examination of the hand specimen prior to thin sectioning. Mineralogy and textural features, and color and weathering characteristics will be noted. Minerals, textures and fabrics may be tentatively classified as primary (e.g. volcanic in origin) or secondary (e.g. deformational/metamorphic). A provisional rock name will be deduced at this stage.

2. Examination of each thin section using a polarizing petrographic microscope. This will include a more definitive identification of each sample's minerals, together with rough estimates of their percentages. Special attention will be paid to minor minerals that may be unique to a specific volcanic flow or other geologic unit, but are too small or sparse to identify in hand specimen. Mineral grain size and preferred orientation will be noted. Other textural features will be noted and described. Again, a distinction will be attempted between those minerals and features that are primary, and those that are secondary.

3. In selected cases, as deemed appropriate, ancillary mineral identification techniques, including X-ray diffraction analysis, may be used.

4. A final, definitive rock name will be assigned.

5. Photomicrographs will be made to accompany the petrographic descriptions submitted.

Summary: Division of Labor

This project involves personnel from six different organizations: Fort Bragg; TRC Garrow, Inc.; the University of North Carolina at Chapel Hill; the Missouri University Research Reactor, University of Missouri at Columbia; North Carolina State University; and the University of North Carolina at Wilmington. Hence it is useful at this point to reiterate the various activities that will be undertaken, emphasizing the division of labor among the organizations.

Fort Bragg

Personnel from Fort Bragg will provide advice and support in all phases of the project. They will be actively involved in choosing the samples for analysis, and in retrieving the actual specimens housed in the archaeological collections on their base. Joe Herbert will be primarily responsible for photographing and describing the ceramic samples. Chris Moore will coordinate the visits to the quarry sites and be responsible for collecting the samples that will be gathered

there. Fort Bragg personnel will also be responsible for compiling the various study reports and results into the final report.

TRC Garrow, Inc.

TRC Garrow will provide overall administration for the project and will conduct reviews of all deliverables prior to transmittal to Ft. Bragg and CERL. They will also be responsible for delivering the powder samples to MURR for neutron activation analysis.

University of North Carolina at Chapel Hill

The Research Laboratories of Archaeology will administer TRC Garrow's subcontract to UNC-Chapel Hill. Vin Steponaitis will coordinate the selection of samples and will act as liaison between TRC Garrow and the various labs in which the analyses will be carried out. RLA staff will provide sherds and rock samples from their collections, photograph specimens as needed, conduct the preliminary sawing of the sherd samples.

Brent Miller, with the help Department of Geology staff, will carry out the preliminary sawing and pulverizing of the rock samples. Splits of his powder samples will be provided to TRC Garrow (for shipment to MURR); and appropriate rock samples will be sent to NCSU (for petrographic analysis). He will also conduct the Nd-isotope study and provide a written report of the results.

University of Missouri at Columbia

Michael Glascock and Hector Neff of MURR will be responsible for conducting the neutron activation analyses of the rock and sherd samples, respectively. They will also produce a report of their results.

North Carolina State University

Edward Stoddard (MEAS) will be responsible for obtaining thin sections of the rock specimens from a commercial lab and carrying out the petrographic analysis of these sections. He will also produce a report of the results.

University of North Carolina at Wilmington

Michael Smith (Department of Geology) will be responsible for obtaining thin sections of the rock specimens from a commercial lab and carrying out the petrographic analysis of these sections. He will also produce a report of the results.

Table 1. Proposed distribution of ceramic sherd samples.

Drainage: *Period*	Sherds (n)	Locality or Site
Cape Fear River:		
Early Woodland	5	Fort Bragg (upland)
Middle Woodland	10	Fort Bragg (upland)
Late Woodland	5	Fort Bragg (upland)
unspecified date	5	McLean Md. or Breeze (valley)
Lumber River	10	Fort Bragg
Yadkin River	10	(site to be chosen)

	50	

Table 2. Proposed distribution of rock samples.

Quarry Group: *Quarry Cluster*	Samples (n)	Rock Type
Uwharrie group:		
Asheboro	5	Uwharrie Complex, rhyolitic tuffs
Western	10	Uwharrie Complex, rhyolite flows (plagioclase)
Eastern	5	Uwharrie Complex, rhyolite flows (quartz-plagioclase)
Southern	5	Uwharrie Complex, rhyolite flows (aphyric)
U.S. 501 group:		
Person County	5	Piedmont Slate Belt metavolcanics
Durham County	5	Piedmont Slate Belt metavolcanics
Chatham group:		
Pittsboro	5	Piedmont Slate Belt metavolcanics
Silk Hope	5	Piedmont Slate Belt metavolcanics
Siler City	5	Piedmont Slate Belt metavolcanics

	50	