

**Documenting subsistence strategies and perishable resource use within Early
Holocene occupations of Northwestern Ontario: Implementing a multi-analytical
approach for the detection of technological variability and subsistence complexities**

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Culture

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ABSTRACT

This thesis presents the results of a micro-analytical analysis, specifically use-wear and residue analyses, on unifacial lithic artifacts from the Electric Woodpecker II (DdJf-12) Early Holocene site, located approximately 25 kilometers east of Thunder Bay, Ontario. The Electric Woodpecker II assemblage consists of a multitude of debitage and artifacts including formal, informal, and expedient tool types with varied morphological attributes. The use of multiple analytical techniques has allowed for the investigation of organic or perishable technologies, the documentation of which is not otherwise possible at most Lakehead Complex sites. The primary goal of this thesis is to determine the function of selected unifacial artifacts from a morphologically diverse lithic assemblage at the Electric Woodpecker II site, and to characterize and identify the presence of organic residues.

The podzolic soil conditions of the Thunder Bay region contribute to the poor preservation of organic remains, limiting the available material evidence in the analysis of lithic artifacts. The interpretations that are possible through macromorphic lithic and spatial analyses can be expanded significantly through the inclusion of micro-analytical techniques. This thesis demonstrates that implementing these techniques within the Thunder Bay region allows for increased documentation of both technological and subsistence complexities. Within this research, use-wear analysis was used to examine the functional uses of a selection of unifacially flaked lithics dating to the Early Holocene period. Use-wear analysis and combinations of residue analysis (microscopic, biochemical, and spectrographic analysis) were used to more fully characterize the proposed residue sources.

A selection of manuscripts submitted to a variety of peer-reviewed journals form the substantive chapters of this thesis. The first of these represents a review of the methodological approaches employed in the study. Several varieties of instrumentation, analytical techniques, and interpretive contexts are discussed in detail, in addition to the benefits and limitations of each. The second and third articles present the results of the use-wear and residue analyses, respectively. The division of these results into two separate publications allowed for a more detailed discussion of each method, specifically as feasibility studies using samples recovered from heavily degraded burial environments. Lastly, the concluding article summarizes the broader implications of the results discussed in articles two and three. An original introductory chapter contextualizes the research discussed here, in reference to current and past trends in use-wear, microscopic, and biochemical residue interpretation. Outcomes of the project include the indication of broad resource use within the region, the use of both generalized and specialized tool types, trends observed within lithic material type selection in relation to tool function, and an unexpected occurrence of hafted expedient tools.

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CHAPTER ONE

INTRODUCTION

1.1 INTRODUCTION

Artifact analyses frequently focus on formal artifacts, where projectile points, scrapers, bifaces and drills are the most commonly analyzed. Expedient artifacts, those crafted with minimal effort and display minimal shaping, are often overlooked. This thesis proposes that expedient or informal artifacts can provide information concerning utilitarian tool use at Early Holocene sites in Northwestern Ontario. To demonstrate this, a sample of unifacially flaked artifacts (ranging from expedient to formal) were selected from the Electric Woodpecker II site (WP II), an Early Holocene site within the Thunder Bay region of Northwestern Ontario. Deglaciation in Northwestern Ontario occurred between 12,000-10,000 years before present (yr B.P.; Lowell et al. 2009). Specifically, the Thunder Bay region became habitable no later than 9380 +/- 150 yr B.P. (Julig et al. 1990; Zoltai 1965). Long periods of fluctuating water levels throughout the final retreat resulted in the formation of several regional end moraines, some of which provided well-drained, raised strandline locations along the shoreline of Glacial Lake Minong, providing abundant seasonal resources and creating attractive habitation areas for the region's earliest inhabitants (Fox 1976; Julig 2002; Kuehn 1998). Primary subsistence models for these occupations emphasize big-game predation with a focus on caribou (Fiedel 1987; Kuehn 2007), or a broader littoral strategy emphasizing seasonal resource adaptation (Julig 2002; Kuehn 1998).

Evidence of resource use among the region’s early inhabitants is scarce; podzolic boreal forest soils rapidly degrade organic materials, frequently preventing their preservation. While several sites have been identified along raised shorelines within the Thunder Bay region (see Table 1.1), the lack of associated organic artifacts hinders inferences about specific resource use and subsistence strategies. Archaeological

Table 1.1 Early Holocene archaeological sites within the Thunder Bay District		
Site Name	Borden	Reference(s)
Biloski	DcJh-9	Hinshelwood and Webber 1987
Brohm	DdJe-1	Hinshelwood 1990; Wright 1963
Cascades Site II	DcJh-37	Arthurs 1986
Crane Cache	DcJj-14	Ross 2011
Cummins	DcJi-1	Dawson 1983; Julig 1984; Julig et al. 1990
Dog Lake Reservoir	Multiple	McLeod 1981
Electric Woodpecker I	DdJf-11	Norris 2012
Electric Woodpecker II	DdJf-12	Gilliland 2012, Gilliland and Gibson 2012; Norris 2012
Electric Woodpecker III	DdJf-14	Gilliland 2012, Gilliland and Gibson 2012; Norris 2012
Mackenzie I	DdJf-9	Gilliland 2012, Gilliland and Gibson 2012; Norris 2012
Mackenzie II	DdJf-10	Gilliland 2012, Gilliland and Gibson 2012; Norris 2012
RLF	DdJf-13	Gilliland 2012, Gilliland and Gibson 2012; Norris 2012
Simmonds	DcJh-4	Arthurs 1986

interpretations are thus limited to analyses of lithic and spatial characteristics, with inferences about other aspects of technology and subsistence deriving from better preserved sites discovered outside of the region. Despite the lack of interpretable macroscopic evidence, over the last decade researchers have begun addressing organic materials at the microscopic level. This is a consequence of the observations that almost

every human action leaves a physical trace, whether in macroscopic or microscopic form (Haslam 2006; Odell 2003). These human actions can be observed and interpreted through various microscopic, biochemical, and spectroscopic techniques.

This thesis demonstrates that micro-analytical techniques involving use-wear and residue analysis can overcome taphonomic limitations common within boreal forest depositional environments. Micro-analytical data in conjunction with the concepts of *artifact as site* and *archaeology of the instant* (Haslam 2006; Loy 1993), significantly augments the information gained from formal, informal, and expedient artifacts (Chapter 1). Interpretations are based on evidence that is located on the artifacts themselves, effectively overcoming common interpretative shortcomings deriving from morphological analysis alone. For example, the manner and direction of use, hafting style, and contact or source material hardness can be determined through detailed use-wear analyses of edge and surface modifications. Interpretation of these are refined and validated using multi-analytical residue analysis. The concurrent use of these approaches provides multiple lines of evidence for the interpretation of tool use and overall subsistence strategy, while increasing the overall interpretative value through evaluations of inter-methodological consistency.

Each methodological approach began with sample selection and the determination of sample size; this is a critical step to evaluate the time and cost requirements of a project. Artifacts were initially screened with a 16x magnification hand lens to tentatively identify tentative working edges of each sample. Low-powered incident light microscopy was then used to confirm these potential working edges and record the presence of visible residues. Working edges were examined a second time after removing the residue

through sonication. On the second examination with low-powered incident light microscopy, the entire working edge was recorded using photomicrography.

Photomicrography protocols included 5-20 images of varying focal depth recorded at approximately 4mm intervals across the working edge. ZereneStacker© imaging software was then used to create composite images of each interval, bypassing the limited depth of field issues common in the microscopic observation of lithic artifacts. High-powered incident light images were also recorded, albeit at targeted areas along the working edges and tool surfaces. Flake scar counts were recorded in three to five locations across each working edge. Feature analysis, the evaluation of polish, striations, rounding, smoothing, stepping, and crushing, was completed on the same images. Inferences of use were based upon both experimental images as well as data available within the current literature. The residue analysis commenced following the finalization of the use-wear analysis. Techniques employed within the approach included high-powered incident and transmitted light microscopy and photomicrography, colorimetric biochemical testing, absorbance spectroscopy, and gas chromatography coupled mass spectroscopy (GC-MS). Each line of evidence was first analyzed and interpreted individually: organic structures observed microscopically were identified when possible, biochemical tests determined the presence of carbohydrates, starches, fatty acids, or proteinaceous molecular compounds, and chemical compounds were identified through GC-MS. Final interpretations were based on a comparison and synthesis of all lines of evidence. A detailed description of the methodology can be found in Appendix A.

The thesis format is based on four manuscripts that have been submitted for publication within refereed journals (see Table 1.2). Due to the concise, targeted format

of articles intended for professional publication, including varying referencing and spelling styles, this introductory chapter offers the necessary background information for a more general readership. The remainder of this chapter presents a brief introduction to archaeological use-wear and residue analysis, and concludes with a detailed description of the thesis organization.

Table 1.2		
Article Submissions		
Chapter	Title	Journal
2	Multi-analytical approaches to lithic analysis: Use-wear and residue	Ontario Archaeology
3	Early Pre-contact use of organic materials within the North Superior Region: Indirect evidence through use-wear analysis	Journal of Archaeological Science: Reports
4	Multi-analytical residue analyses on Early Holocene lithic assemblages within the boreal forest of Canada: A feasibility test and an evaluation of residue interpretations	Journal of Archaeological Science: Reports
5	Early Holocene subsistence variability within Northwestern Ontario: Incorporating lithic use-wear and residue analysis for the detection of perishable technologies	American Antiquity

1.2 Background

1.2.1 Use-wear Analysis

While interpretation of tool function has long been part of archaeological investigation, serious attempts to infer these function based on microscopic use-wear did not take place until the mid-1900s (Odell 2003). Sickle gloss, an easily discernable surface polish, was the first wear to be considered primarily due to its unmistakable appearance on sickle blades (Curwen 1930, 1935). Serious attempts to identify use-related wear with magnification on other types of tools were not noted until 1957, due in

large part to the publication of Sergei Semenov's seminal work *Prehistoric Technology* (Semenov 1957). The English translation was introduced to western archaeological scientists in 1964 (Semenov 1964), triggering an initial period of widespread implementation of use-wear analysis in North America.

During early implementation of use-wear studies, debates arose concerning the effectiveness of low (<100) and high (>100) powered magnification. Other challenges included the supposed lack of reproducibility and quantification of results, and standardized terminology. This resulted in underutilization of the technique until the last decade, in which there has been a resurgence of interest due in part to effective evaluations of the feasibility and scope of the technique. Additionally, researchers have access to more accurate and sensitive instrumentation, improved photomicrographic techniques, increased experimental comparative collections within the literature, and an increasingly standardized terminology. Current research ideology supports the use of multiple lines of evidence for the accurate interpretation of tool function.

Four main approaches are currently used in the determination of use-wear damages; low and high powered incident light, confocal laser scanning, and scanning electron microscopy. Although the scale and resolution of each approach differs greatly, they all enable documentation and analysis of edge and surface damages or modifications related to use. The creation of comparative databases, in addition to more numerous experimentally re-created comparative collections, has provided a growing basis for current analysts to aid in use-wear interpretation.

1.2.2 Residue Analysis

Organic residue analysis has recently emerged as a technique applied to archaeological materials. It includes a multitude of methods, many of which are already utilized in biomolecular and forensic studies as well as organic and analytical chemistry. When applied to archaeological specimens, the techniques are used to determine the nature and origin of organic residues that are otherwise unanalyzable because of their amorphous nature, or their degradation prevents their identification or characterization using traditional techniques (Evershed 2008). The preservation of these materials is determined by a multitude of variables, discussed further in Chapter 4.

Lithic materials were long considered to be unsuitable for residue analysis due to the lack of protection offered by non-porous, silica-rich materials (Evershed 2008). However, in recent years the use of increasingly sensitive instrumentation has demonstrated that lithic materials do have the capacity to preserve residues. While the preservative capacity of these materials is partially determined by the burial environment, the use of multi-analytical techniques has demonstrated that samples from highly degradative environments may still yield positive results (Bouchard 2016; Cook 2015; Newman and Julig 1989; Matheson and Veall 2014).

1.2.3 Background Summary

The concurrent use of these techniques is a relatively recent development (Bicho et. al 2015). Methods include low or high powered use-analysis in combination with incident or transmitted light microscopy, biochemical testing, Fourier transform infrared spectroscopy (FTIR), enzymatic digestions, and gas-chromatography coupled mass spectroscopy (GC-MS), amongst others. Studies have demonstrated that the use of a multi-analytical approach maximizes the available data from degraded archaeological

materials. The recovery of this information is invaluable in the interpretation of past resource use, and provides visibility to what is otherwise invisible within Early Holocene assemblages in boreal forest depositional environments.

1.3 Terminology

Effective comprehension of this thesis requires an understanding of the terminology employed within use-wear and residue analysis. This section provides a brief introduction, while key concepts and specific terms are described more fully in Chapter 2.

Use-wear analysis primarily seeks to determine tool function, although low and high powered microscopy is also informative of source material hardness and other characterizing features. The tendency of silica-rich materials to fracture conchoidally allows interpretation of the direction, force, and motion required for such wears to occur. Post-depositional or taphonomic damages can mimic those related to use, and can only be separated with a detailed analyses of wear distributions, usually with reference to an adequate comparative collection created through experimental studies. It is important to note that the variety of ways in which an artifact may have been used directly (e.g. in a specific task) or indirectly (e.g. storage or transportation) are numerous; creating a comparative sample of known wears that include all possible variables is neither time or cost efficient for most researchers. As such, a detailed review of comparative samples within the literature is also required.

Residues are broadly defined as materials that have been left on a tool surface or edge as a consequence of the latter being used to process that material (Kooyman 2000). These can vary from macroscopically visible amorphous residues (e.g. hafting adhesive)

to nearly invisible compound structures ranging from starch granules to feather barbules. The placement of residues can reflect intentional (e.g. tool hafting or binding) or incidental activities. The latter typically develop as a by-product of functional activities, depositional context, or post-excavation cross-contamination. Residue observations can be made *in situ* with incident light microscopy, which requires minimal preparation, or via extraction with transmitted light microscopy. Extraction solvents or solutions are selected based on characteristics of the molecular compounds the study is targeting. Polarity and solubility characteristics of molecular compounds vary, as do those of common extraction agents. The effects of these traits on extraction solution selection are discussed in detail in Chapter 4.

The preservation of residue on artifacts deposited within podzolic boreal forest depositional environments remains largely unexplored. Apart from tentative blood residues (Newman and Julig 1989) and recent, unpublished work from Lakehead University (Bouchard 2016; Cook 2015), little research of this kind has been completed to date. Taconite, the Gunflint Formation material most heavily utilized at documented Early Holocene assemblages within the Thunder Bay region, is an iron-rich silicate consisting of moderately cemented granules within a silica matrix. The porosity of this material along with its natural tendency to form covered, stepped stress fractures (from use and non-use related stressors) may provide a sort of protective coating for potential micro-residues. Increasingly sensitive and accurate instrumentation can detect these compounds, despite their lack of visibility using other techniques. The lack of visible residues within the current study resulted in the use of microscopy, biochemical testing,

absorbance spectroscopy, and GC-MS techniques in order to provide numerous lines of evidence.

1.4 Sampling

The sample of this project was the result of the available site catalogue inventory. The cataloguing of Electric Woodpecker II materials is ongoing, preventing a full assessment of the assemblage at the time of this writing. Preliminary cataloguing and sorting of materials resulted in a selection of ‘pulled tools.’ To select the sample described here, each unifacially flaked tool was examined to determine the likelihood of use via macroscopic edge damage. All but two of the selected artifacts are made of dark grey to red taconite; the remaining two samples of Gunflint Formation banded cherts. A detailed discussion of the lithology of these samples is outside the scope of this thesis, and will therefore not be discussed in greater detail.

The final number of samples examined differed for each approach. Thirty-two artifacts were analyzed in the use-wear analysis, while only 22 samples were included within the residue analysis. The latter sample includes those with the clearest results deriving from the GC-MS analysis, a key method within the residue analysis. In order to complete an in-depth comparison, synthesis, and discussion of all the methods employed, the final sample size is limited to the 22 included in the residue analysis (Fig. 1.1).

1.5 Thesis organization

The papers are formatted sequentially from review (Chapter 2), to results (Chapters 3, 4), to discussion and interpretation (Chapter 5). The formatting of each chapter reflect the requirements specified by each journal, and will therefore differ from

chapter to chapter. However, minor concessions have been made to these requirements in order to improve the consistency of the overall thesis. These concessions are limited to

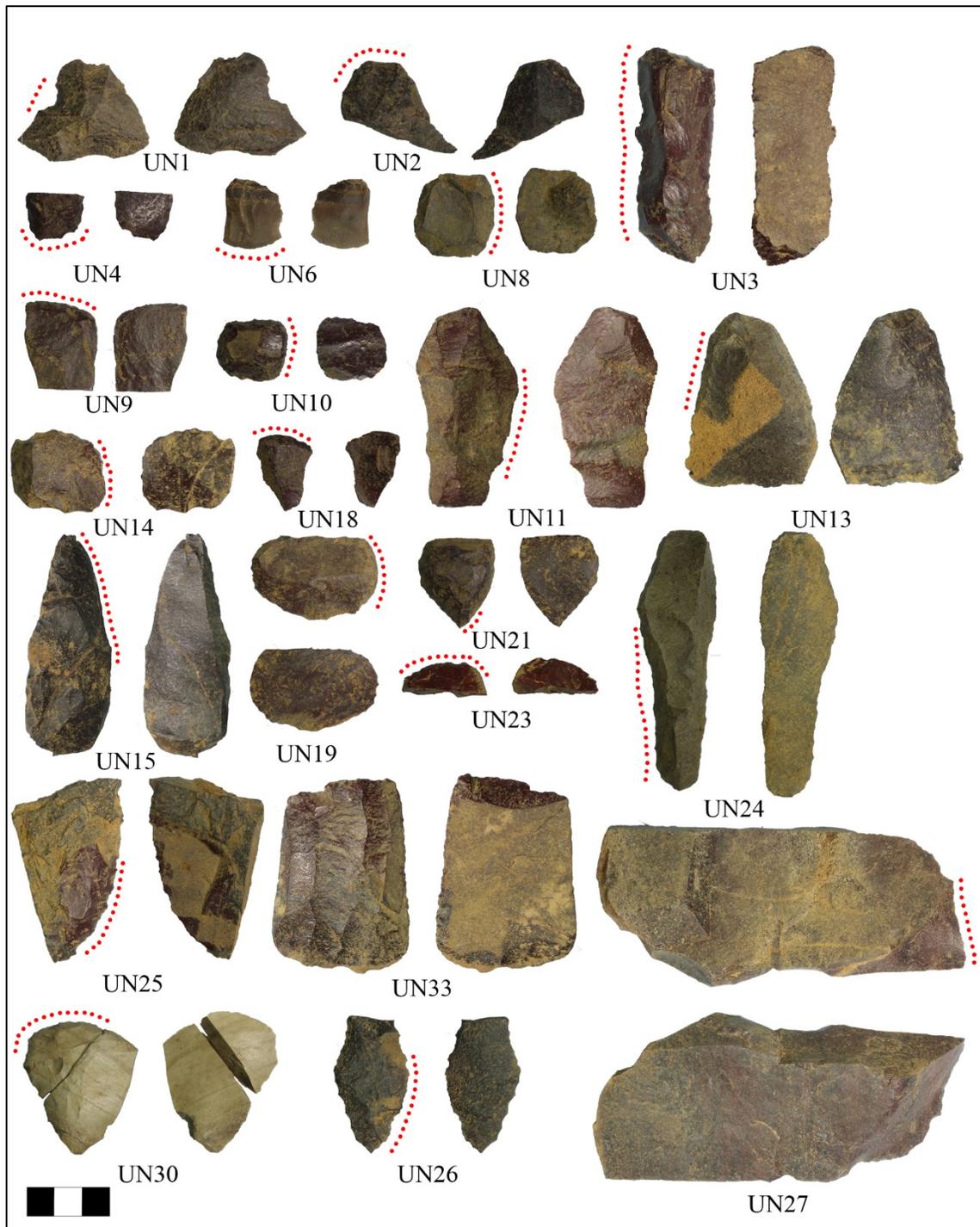


Figure 1: Dorsal and ventral images of final sample.

margin size and the elimination of line numbering. Due to these differences in formatting, the spelling, references and the table of contents appear different than those in standard theses. Rather than an exhaustive list upon the conclusion of the thesis, references specific to each chapter will be provided at the end of that chapter. Tables are located immediately after the conclusion of each chapter. For ease of location, all sections have been numbered sequentially in the main table of contents. Individual chapter introductions preceding each chapter have been provided in order to aid in the cohesion of the thesis.

Chapter two presents a preliminary guide for novice analysts on the basic approaches to multi-analytical lithic research involving use-wear and residue analysis. The chapter is divided into several sections, beginning with research scope, followed by information concerning sample selection, and a detailed discussion concerning the collection, analysis and interpretation of data. Several methodological approaches are discussed, including the four broad streams of use-wear analysis currently practiced (low and high powered, confocal laser scanning, and scanning electron microscopy), as well as several approaches to residue analysis (microscopic analysis, biochemical testing, absorbance and gas chromatography coupled mass spectroscopies). As such, this preliminary chapter serves as a review of existing literature and methodological approaches.

Chapters three and four present the results of the use-wear and residue case studies. Chapter three discusses the results of the use-wear portion of the project and its implications toward technological variation and specialization at the WPII site. Tentative results concerning faunal and floral resource exploitation were obtained, in addition to

evidence of hafting on both formal and informal artifacts. Chapter four discusses the results of the residue analysis. Source materials were successfully identified although limited to fairly basic designations: plant, animal, bone, wood, pitch, and burnt organic matter. Observations concerning technological variations and specialization or generalization first discussed in Chapter 3, were echoed within the additional lines of evidence pursued throughout the residue analysis. Given the preservative limitations of the study area, the analysis was conducted as a feasibility test in addition to providing information about source materials. A Kruskal-Wallis statistical analysis was used to determine the presence of statistically significant relationships between the selected methods. Based on these results, recommendations concerning high data yielding combinations of techniques are provided.

Chapter five synthesizes the results of both methodological approaches into a final, cohesive interpretation supported by multiple lines of evidence. Resulting implications including evidence indicative of faunal, floral, avian, and aquatic resources are discussed in relation to regional subsistence models. The study revealed an unexpected frequency of hafting styles overall, specifically in relation to informal artifacts. An argument toward the increased use of these micro-analytical methods within regions with similar geographic limitations for the documentation of otherwise unquantifiable complexities in Early Holocene assemblages is presented. This final chapter provides conclusions and suggests possible future directions for the use of these techniques within boreal forest burial environments.

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CHAPTER TWO

MULTI-ANALYTICAL APPROACHES TO LITHIC ANALYSIS: USE-WEAR AND RESIDUE

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CHAPTER INTRODUCTION

This chapter serves as a review of existing literature concerning use-wear analysis and multi-analytical residue analyses. In addition, it provides a review of methodological approaches for each technique. The focus of this paper is to provide information necessary for the implementation of these approaches into broader analyses and interpretation. The formatting is broken down into several sections.

First, the background of each approach is provided to contextualize the use of these techniques within modern archaeological science. Second, research scope and inherent limitations to the approaches are discussed. Due to these inherent limitations, sample sizes are often limited. To counter this, brief descriptions of multiple theoretical approaches pertaining to small samples sizes are provided. Third, sample preparation is discussed in detail, including common cleaning techniques for use-wear analysis, extraction techniques for residue analysis, and the combination of the two into a single phase as was used within this study. Data collection is the fourth stage discussed, in which microscopic approaches to use-wear analysis and several approaches to residue analysis are described in detail. The benefits, limitations, protocols, and basic information concerning instrumentation are included. Data analysis, the fifth stage, provides

descriptions of terminology and key concepts necessary for both use-wear and residue analysis through a detailed written description, photographs, and hand-drawn diagrams. Several categories of residues commonly identified on archaeological specimens are likewise described. The final stage, data interpretation, discusses categorical interpretations commonly included within use-wear and residue studies. The identification of source material hardness, manner of use, and style of grip are described within the use-wear section. An equally detailed discussion of residue interpretation is beyond the scope of the research presented here, and as such has not been included. Instead, the basic interpretive process is described. This process consists of six key determinations, and remains the same for all residue analyses. An additional section describing additional factors that affect the determination of function and source material identification is included. This final section provides a brief overview of common limitations experienced during the interpretive process of each approach, as well as methods used to overcome them.

Submission Title Page: Multi-analytical approaches to lithic analysis: Use-wear and residue

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Multi-analytical approaches to lithic analysis: Use-wear and residue

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This paper represents a preliminary guide for novice analysts interested in utilizing approaches involving the analysis of use-wear and residues on lithic assemblages. The approach is particularly valuable for assemblages which lack organic components due to destructive burial environments or samples which lack contextual information, and has considerable potential to aid in the reconstruction of past human behaviors. The four broad streams of use-wear analysis, low and high-powered optical microscopy, confocal laser scanning microscopy, and scanning electron microscopy, are described. Several approaches to residues analysis are provided as well, and include both transmitted and incident light microscopy, scanning electron microscopy, biochemical testing, absorbance–spectroscopy, and gas chromatography coupled mass spectroscopy. While not exhaustive in nature, steps including sample selection and cleaning, as well as data collection, analysis, and interpretation are discussed. The limitations and benefits of each are provided throughout.

1.0 Introduction

Past populations maximized their available resources through flexible resource exploitation, resulting in sustainable relationships between populations and their environment (Fagan 2008). These relationships and strategies are recognizable within the archaeological record through preserved material artifacts, reconstructed paleoecological data, and a host of increasingly accurate analytical techniques and methodological approaches. In archaeological contexts lacking organic artifacts or stratigraphic differentiation, methods which maximize interpretable data from lithic artifacts become increasingly important. Understanding the function of these tools is integral in the interpretation and understanding of site occupants (MacDonald 2014). Two of these approaches, use-wear and residue analysis, have proven to be powerful tools in the functional analysis of stone tools and the documentation of past resource exploitation (Odell 2003).

The extensive analysis of microchipping and micro-feature formation

on the working edges and surfaces of artifacts provides a means to infer the use of perishable technologies otherwise invisible within an archaeological assemblage (Loebel 2013; Miller 2014; Soffer 2004). It has been demonstrated that these types of damages relate directly to both the manners of use and the materials that were processed (Keeley 1980; Lawn and Marshall 1979; MacDonald 2014; Odell 1979; Tringham 1974). Methodological approaches incorporate a minimum of one of four techniques: optical microscopy in low (<100x) or high (>100x) magnifications, confocal laser scanning microscopy (CLSM), or scanning electron microscopy (SEM). In recent years, multi-analytical residue analysis has been utilized in conjunction with use-wear approaches to provide increasingly detailed results (Bicho et al. 2015). Approaches addressing residue analysis range from fairly simple microscopic analysis and biochemical testing to increasingly complex spectroscopic and chemical analyses (see Section 5). The combined, multi-analytical approach has been shown to successfully demonstrate lithic tool function through the interpretations

of contact material hardness and manner of use with increasingly specific identification of contact materials through residue analysis (Briuer 1976; Soffer 2004).

The following article does not represent an exhaustive review of available methodological approaches, procedures, or interpretive styles. Rather, it acts as an introduction to multi- and micro-analytical lithic analyses. The capacity of each instrument, sample requirements, methodological protocols and procedures, and the benefits and limitations of each method are provided. This is followed by a discussion of data analysis and subsequent interpretation of results. These sections provide descriptions of common wear patterns and organic compounds as well as an interpretive guide to tool function. The early history and development of each of these fields is extensive and merits its own discussion; therefore, it is not explored within the scope of this paper. Readers are referred to the reference list for further study.

2.0 Background

Microwear analysis is based on the observation that the use of a stone tool in different motions and on different materials will result in distinctive, interpretable patterns on the working edge (Keeley 1974; Miller 2014; Odell 1975; Semenov 1964, Tringham 1974). It involves the microscopic examination of artifact edges and surfaces through varying levels of magnification in order to determine the manner in which an artifact was used. Additionally, microwear analysis allows for the differentiation of damage patterns caused by manufacture and post-depositional taphonomy from those directly related to use (Adams 2014).

The field experienced a rapid period of growth in the 1970s (Ahler 1979; Hayden 1979; Keeley 1974; Odell 1975; Tringham 1974), followed in the 1980s by a period of

criticism based on the low reproducibility of results, the lack of standardization of terminology, and an overly ambitious scope of research (Odell 2003). The acknowledgment of inferential limitations of the methodology was addressed through improved photo-documentation, increased quantification, a growing standardization of terminology, as well as the increased use of blind testing to determine the reliability of interpretations. These developments allowed for the growing acceptance of the technique in functional analyses (Van Gijn 2014). Today, microwear analysis has become more widely accepted as an indicator of prehistoric technological and economic processes (Hamon 2008; Wiederhold and Pevny 2014).

The microscopic examination of lithic artifacts aids in the detection of wear patterns that are not visible through macroscopic observation alone, and aids in the avoidance of interpretive biases due to preconceived notions of tool use based on morphology (Van Gijn 2014). The development and standardization of varied methodological approaches has continued in recent years through the use of experimental studies (Jennings, 2011; Lerner 2014; Miller 2014), blind tests (Rots et al. 2006; Stevens et al. 2010), classification and quantification techniques (MacDonald 2014; Stemp 2014), and the addition of residue analysis (Langejans and Lombard 2015; Marreiros et al. 2015).

Organic residue analysis utilizes techniques employed in both microscopic and chemical analysis to identify the nature and origins of unknown organic remains which cannot be characterized with traditional techniques due to their amorphous form or degradation to the point of invisibility (Evershed 2008). The preservation of these residues is the result of chemical reactions caused by the heat and friction that occur between lithic and source material (Marreiros et al. 2015). The quality

of the preservation is determined by a multitude of variables: moisture levels of the source material, the percentage of silica in both the stone tool and the source material, the acidity or abrasiveness of the burial environment, the chemical composition of the artifact, the porosity of the artifact, and the level of protective coatings developed over time (Hardy 2004; Levi-Sala 1986; Lombard 2008; Loy 1983; Shanks et al. 2001). Ceramic vessels have proven ideal at preserving organic residues, both within the mineral matrix of the vessel itself, as well in carbonized food residues (Evershed 1993; Malainey 1999c).

Until recently, lithics were deemed generally unsuitable for organic residue analysis due to the lack of protection offered by the non-porous minerals (Evershed 2008). However, recent research has demonstrated that through a multi-analytical approach with increasingly sensitive instrumentation, lithic materials do have the capacity to preserve residues, even within podzolic soil conditions (Bouchard *n.d.*; Cook 2015; Hodgson 2016a; Lombard 2006; 2006a; Newman and Julig 1989). Micro-fractures, cracks, striations, and varying degrees of porosity within specific lithic types act as a protective coating, increasing the quality of preservation (Haslam 2006b; Loy 1983). Previous studies have focused on blood and muscular tissue (Prinsloo et al. 2014), lipids and fatty acids (Evershed 1993), bone, scales, collagen, and hair (Robertson 2002; Stephenson 2015;), plants and microfossils (Crowther 2009; Fullagar 2006; Pearsall 2004), and pigments (Lombard 2006a). Numerous spectrographic and chemical approaches have been utilized in the analysis of molecular and isotopic compounds within residue.

3.0 Scope

Research question and scope should be focused and narrowed if necessary prior to initiating micro-analytical project. For example, if the analyst is primarily interested in how an artifact was used, then use-wear analysis may be the ideal focus. Should specific resource exploitation be the focus, then residue analysis would be more appropriate. For projects attempting to answer both of these questions, the time required for multiple types of analytical and methodological procedures should be taken into account, and sample size adjusted accordingly. As is common with every archaeological investigative technique, neither use-wear nor residue analysis has the capacity to define the full picture of past lifeways. Even though increasing observations have been recorded in numerous experimental studies, more reference databases created, and many blind tests completed within each approach, the simultaneous utilization of both approaches assures a more complete compilation of interpretable data.

Due to the time investments required for a multi-analytical approach, it may not always be appropriate to analyze the large sample sizes typical of traditional methodological approaches (e.g. spatial, typological, or debitage analyses). The concepts of *artifact as site* (Loy 1993) and *archaeology of the instant* (Haslam 2006b) represent alternate theoretical approaches appropriate for small samples sizes.

3.1 *Artifact as Site Concept*

The artifact as site concept effectively creates a situation in which an individual artifact can be as informative as a larger site, albeit in a different way. The approach is ideal when interpreting a limited sample size which would otherwise be inappropriate for more traditional analysis. The concept focuses on the use of micro-analytical techniques to maximize useable data from the

artifact, in essence creating a micro-scale ‘site’ consisting entirely of the observable and interpretable data from the tool (Loy 1993). It should be noted that without a culturally diagnostic artifact in the study or a single component site, limitations will remain concerning the artifact’s interpretation, context, and placement within a morpho-chronological history.

Numerous micro-analytical techniques are suited to this approach, providing an ideal basis for a multi-analytical approach. Small-scale inferences concerning subsistence strategies, perishable technologies, and resource use are possible using this approach, although the small sample size limits the broader scale implications of any findings. Relevant information can be gained from single-approach techniques, but without additional supporting evidence, caution is required during the interpretation and broad-scale application of the results.

3.2 *Archaeology of the Instant*

Like the artifact as site concept, archaeology of the instant concept was developed and is best suited for small or very small samples. The approach provides a way of communicating the results of small-scale analyses of specific actions that occurred at a specific moment in time (Haslam 2006). The greater the sample size, the greater the number of interpretable moments, or instants. It involves notions of narrative, scale, action, and agency as a way of expanding the theoretical scope and application of residue studies. The detail provided through this approach brings the audience ‘face to face’ with the narrative of prehistory in a way that more generalized macro-scale discussion cannot (Roe 1980:107), and provides a way in which individual behaviors can be clearly communicated and relatable to modern readers (Cahen et al. 1979).

4.0 Sample Preparation

Prior to any analysis, overview photographs and/or drawings of the ventral and dorsal surfaces of each artifact must be produced. Cross-sections of the artifacts demonstrating thickness, curvature or other features may also be necessary. All quantitative measurements (e.g. length, width, thickness, edge angle, etc) should be documented in this early phase (Tringham 1974). If preliminary observations with low magnification indicate damaged edges or surfaces, a targeted study area may be selected. These targeted areas may change with subsequent stages of analysis.

The time requirement for each combination of multi-analytical techniques varies. Ideal sample sizes remain relatively small, but can be adjusted for each combination. The time required for edge cleaning or residue extraction must be taken into account when planning the study, as further analysis is dependent on this first critical step. When chemical residue analysis is being completed simultaneously, this step can be condensed into the residue extraction phase.

4.1 *Cleaning*

Clean study areas are mandatory to complete an effective use-wear analysis. Numerous approaches exist in the literature and are dependent on the methodological approach taken by the analyst. In early use-wear studies, the cleaning solution was typically discarded. However, with the incorporation of residue analysis it is increasingly common to use organic solvents as cleaning agents, thus creating a usable residue extraction as a byproduct of the cleaning process (Bouchard *n.d.*; Hodgson 2016b).

Cleaning practices can affect the surface texture of the artifact, reducing the visibility of quantifiable traces of use-wear and subsequent interpretation (Evans and

Donahue 2005). While a fully cleaned edge is ideal for analysis, it may not be appropriate for all samples; for example, samples which are fragile in nature or which may require additional research in the future. In these circumstances, a less invasive approach may be preferable.

Cleaning is typically conducted by soaking or sonicating the artifact. Distilled water with a mild detergent is frequently used to remove sediments, residues, or post-excavation contaminants. A second common approach requires a total or partial submersion in distilled water with or without detergent, and then followed by short alternating soaks in both acidic and basic solutions (Evans and Donahue 2005; Keeley 1980). Spot removals, a technique already employed in residue analysis, may prove applicable in the cleaning of small, discrete locations. The applicability of small-scale cleaning is highly dependent on the porosity of the lithic material and may not be suitable for all artifact types. While detailed cleaning protocols will not be discussed further here, procedures used in other studies will be referenced as part of Table 2, located at the end of the publication.

4.2 Residue Removal

All living systems consist of organic compounds. These include carbohydrates (sugar, starch, etc.), fatty acids (fat, oil, wax), and proteins (amino acids) amongst others, with specific biomarkers sometimes known for family, genus, or species-specific identification (Loy 1997). Solvents chosen in archaeological residue analysis are often determined by what the researcher is expecting to find on any given tool (Evershed 2008; Loy 1997; Pearsall et al. 2004). For example, a tool with wear indicating a cutting use is more likely to have residues consistent with fatty acids, proteins, or starches, while an artifact used for grinding is likely to have a much higher ratio of carbohydrates and starches.

Utilizing a variety of solvents increases the range of archaeological residues, or solutes, which can be extracted and interpreted (Evershed 2008; Loy 1997; Pearsall et al. 2004). Characteristics that can be investigated include polarity, solvent binding, boiling or melting points, densities, or relative permittivity (Crowther et al. 2014). Emphasizing the polarity and preferential binding of biochemical solvents has proven valuable in the investigation of mixed, unknown archaeological residues that occur due to the tendency of organic materials to become increasingly polarized and experience altered binding mechanisms over long periods of time (Crowther et al. 2015). Extractions are taken by submerging an artifact in a specific chemical solution (see Table 1) for a pre-determined period of time, typically five minutes to one hour. The residue solution is then desiccated to a desired volume to avoid dilution, and stored according to the selected methodological protocol. When completing use-wear and residue analysis concurrently, it is possible to complete the artifact edge or surface cleaning via sonication with a specific chemical solution. Combining the steps allows for an efficient use of materials, with as little exposure as possible to the artifact.

4.3 Limitations

Cleaning and extraction restrictions are typically due to the physical limitations of the artifact, or due to the introduction of contaminants into the residue solution. Arguments have been made concerning the use of plastic extraction vessels, prohibiting their use within this stage of analysis (Crowther et al. 2015). Sterile glass vessels are used instead, but appropriately sized ones

Table 1: *Organic solvents used in residue extractions**

Archaeological	Ratio	Compound	Reference
Chloroform/Methyl Esters	N/A	Fatty Acids, various	Mazzia and Glegenheimer 2014
Chloroform/Methanol	2:1	Fatty Acids	Copley et al 2005
Chloroform/Methanol	2:1	Fatty Acids, Beeswax	Evershed et al 2003
Chloroform/Methanol	2:1	Cholesterol	Stott and Evershed 1996
Dichloromethane		Resin Acids	Ribechini et al 2008
Ammonium hydroxide		Amino Acids	Barnard et al 2007
Acetonitrile		Fatty acids	Barnard et al 2007
Dichloromethane/methanol	1:1	Resin Acids, Fatty Acids	Charrie-Duhaut et al 2007
Dichloromethane/methanol	1:1	Resin Acids	Regert et al 2008
Dichloromethane/methanol	2:1	Resin acids, Fatty Acids	Reviewers comment
Dichloromethane		Resin Acids	Stern et al 2003
Dichloromethane			Hogberg et al 2009
Methanol/water/acetic acid	9:9:2	Polyphenols	Romanus et al 2009
Methanol		Resin Acids	Findeisen et al 2007
Chloroform/methanol/citrate buffer	1:2:0.8	Various	Fbuonasera et al 2005
Acetonitrile/ethanol/water	1:1:1	Various	Crowther et al 2015; Hodgson <i>n.d.</i> ; Bouchard <i>n.d.</i>
Non-archaeological			
Methanol/water	19:1	Resin acids	Bohme et al 1997
Chloroform/methanol	2:1	Fatty Acids	Michalski et al 2013
Ethanol/water	19:1	Resin Acids	Cheng et al 2013
Chloroform		Resin acids	Fukuda et al 2006
Acetone		Resin acids	Ferreira et al 2001
Ethanol/water		Resin acids	Malarvizhi and Ramarkrishnan 2011
Acetone		Alkaloids	Darby et al 2001

**Modified from Crowther et al 2015*

may not be readily available. Post-depositional contaminants from storage, handling, or airborne particles may be introduced into the residue solution from the artifact, lab, or field environments. Additional contaminants may be present from conservation, restoration, or fumigation practices. While the contaminants can be ruled out through a detailed chemical analysis or comparative microscopy, they add challenges to archaeological chemistry

not seen in more routine analytical applications (Pollard et al. 2007).

5.0 Data Collection

Approaches to use-wear and residue analysis continue to develop as both technology and instrumentation become increasingly accurate and accessible. These approaches vary from relatively simple low-

or high-powered optical microscopic analysis (10x - 500x magnification), to higher magnifications from laser and electron sources (>1000x). Incident light microscopy allows the characterization of *in situ* residues and wear patterns. Transmitted light microscopy requires the residue be placed on a glass slide, and allows for the examination of microscopic compound structures without the depth of field issues common to *in situ* observations. Non-optical magnification such as CLSM and SEM allow for an even greater magnification and increasingly detailed characterization of diagnostic compound structures.

These methods represent the four most commonly employed microscopy methodologies within use-wear analysis, and can be equally applied to residue analysis (excluding CLSM). Approaches pertaining specifically to residue analysis adopted from other fields of study (biomedicine, chemistry, etc), have continued to grow in their applicability to archaeology. These methods range from broader characterization techniques (biochemical testing, absorbent light spectroscopy, etc) to increasingly complex analysis at molecular or isotopic levels (GC-MS, FTIR, etc). The method(s) selected are dependent on the specific research questions being investigated, and each approach is subject to different strengths and weaknesses. The following sections discuss these factors, along with the technical aspects of each approach. While this is not an exhaustive list, it provides a short introduction to several commonly used techniques. Additional readings are found within the references, and short summaries of studies utilizing each method can be found in Table 2, located at the end of the publication.

5.1 Experimental

Use-Wear Analysis. Experimental studies and the quantification and standardization of methods play an important role in use-wear and residue studies (Marreiros et al.

2015:10). These studies familiarize the analyst with different variables that affect the formation of wear on tool edges and surfaces, in addition to the effects of intra- and inter-material variability (Bradley and Clayton 1987). Creating a collection of function-related wear patterns builds a comparative database and aids in the differentiation between post-depositional and use-related damages. Previous studies have proven invaluable for the interpretation of archaeological wears, and have often demonstrated that form does not necessarily follow function (Ahler 1970; Odell 1979; Tringham 1974).

Experimental studies fall into two categories: prescriptive or reactive. Prescriptive analyses employ a broad approach and are typically completed prior to the analysis of archaeological specimens. Varying functions are completed, typically including cutting and scraping in a variety of manners, as well as whittling, chopping, and drilling, amongst others. A multitude of organic materials are used throughout the activities, and both material and activity types are dictated by regional availability and the tool type being investigated. The experiments may involve several stages of use with photo-documentation of processual wears. Conversely, documentation may be limited to pre-use and completed task stages. Reactive studies are typically, but not always, a secondary addition to the preliminary experiments. They represent a more targeted approach with increasingly narrow research questions, usually as a result of preliminary observations. Rather than producing a variety of wear patterns within a comparative setting, this approach focuses on one specific motion to determine if archaeological wear patterns may have been caused in a very specific fashion. Source, or contact materials, are tested simultaneously in either approach to investigate the effect of varying hardness and elasticity on the accrual of wear over time.

Many use-wear analysts build and utilize their own comparative collections (Keeley 1976, 1977a, 1977b; Newcomer and Keeley 1979; Tringham et al. 1974).

Residue Analysis. Experimental residue analysis is based on the same premise as that of use-wear: to create an experimental reference collection. While particularly pertinent to incident and transmitted light microscopic approaches, it can also be applied to chemical analyses. The aim is to examine similarities and differences in residues extracted from modern organic materials with their unknown archaeological counterparts. Reference collections can be created in several ways: specific organic materials can be processed directly, or replica artifacts may be used in specific tasks on selected materials, and then analyzed in the same approach as the archaeological specimens. In these ways experimental databases for both wear patterns and residues can be tailored specifically to suit the specification of the investigation.

Limitations. Perhaps the biggest limitation to experimental analyses is acquiring adequate testing materials. While most source materials (meat, hide, wood, etc), are fairly easy to acquire, replica tools may prove to be more difficult. Unless the researcher is a skilled flintknapper, acquiring an adequate number of flakes and tool types may be costly. Testing for an adequate number of variables (see Table 3) rapidly increases the time required to complete what begins as a fairly simple analysis, particularly when documenting processual wear requiring cleaning and observations at multiple intervals. Additionally, each methodological approach has specific limitations that will need to be considered; these will be discussed in the following sections.

5.2 Optical Microscopy

Use-wear. The use of both low- and high-powered magnification in the analysis of use-related damages provides the greatest amount of interpretable data concerning tool function (Odell 2001). Edge damages including scar morphology, polish, and other types of micro-features visible with magnification under 100x. High-powered magnification (>100x) allows increasingly detailed descriptions of micro-scarring, polish, and striation formation (Marreiros et al. 2015:10). The documentation of wear damage in this manner overcomes the influence of preconceived notions of tool use within the study (Van Gijn 2014).

Edge angle, profile, damage, damage distribution, and diagnostic fractures are the primary focus of study with the low-powered technique (Kamminga 1982). The use of blind tests has indicated that increasingly specific functions may be interpreted with increasing levels of experience (Newcomer and Keeley 1979). Novice or amateur analysts should limit inferences to the manner and direction of use and the hardness of the contact material (Grace 1996; Keeley and Newcomer 1977; Odell 1980). The completion of preliminary low-powered microscopy is crucial in avoiding excessive washing or the use of damaging solvents that could unintentionally alter or remove analytically diagnostic residues (Van Gijn 2014).

High-powered microscopic analysis was introduced in North America by Lawrence Keeley (1980), and used incident light microscopy ranging from 100x to 400x magnification. With this higher powered method it becomes possible to not only determine the hardness of the contact material, but also identify and classify different types of materials with increasing confidence (e.g. hide, wood, bone, antler; Keeley and Newcomer 1977). It was during this time period that the field experienced increasing experimentation and

Table 3. *Variables to consider during experimental use-wear analysis*

Variable	Attributes	Characteristics
Scars	Initiation	Bending, flat
	Shape	Scalar, triangular, trapezoidal, rectangular, half moon
	Termination	Feather, step 1, step 2, hinge, snap
	Size	0-4000um (dependent on scale)
	Distribution	Continuous, discontinuous, aligned, isolated
Features	Polish	Glossy, matte, greasy, bright, mixed
	Polish distribution	Continuous, discontinuous, patchy
	Striations	Length, width, terminations, direction
	Nibbling	Size of crenellations, degree of concavity rounding, distribution
	Smoothing	Invasiveness, light to heavy extent, distribution
	Rounding	Invasiveness, light to heavy extent, distribution
	Crushing	Light to heavy extent
	Crazing	Extent, presence of other heat indicators
	Stepping	Extent, shape of scars, location, distribution
	Snap fractures	Shape, location, distribution
Variables	Direction of use	Longitudinal, transverse, circular, bidirectional, unidirectional
	Source hardness	Soft, medium, hard
	Source elasticity	Low, medium, high
	Source freshness	Fresh/raw, cooked, dried
	Grip	Acute or perpendicular Hafted or handheld

documentation of the formation and classification of polishes (Vaughan 1985).

Light source placement varies with the microscopic approach. Incident light microscopy (stereoscopic) has the light source located directly above the artifact. The artifact itself can be placed at different angles to allow for a shadowing effect, at times making subtle features or microtopographic changes increasingly discernable. All edges and surfaces are systematically analyzed in order to record small features and fractures, and more discrete areas are selected for

further high-powered microscopic investigation (Kamminga 1982; Odell 1979; Tringham 1974). Photostacking software can be used with both methods to combine images from multiple focal planes. Transmitted light microscopy, in which the light source is located below the specimen, is not applicable to use-wear analysis and will be discussed in the following section.

Residue. Residues can be observed with magnifications under 100x, but typically analyses require higher magnifications to

fully characterize and interpret residues. Amorphous residues in particular are limited to more powerful microscopic imaging. Visual identifications of *in situ* or extracted residues should be interpreted with caution; without additional lines of evidence, it is difficult to irrefutably confirm the source of a residue based on morphological structures. This is particularly true for the novice analyst. The choice of light source is dependent on the context in which the residues are observed: *in situ* with incident light, or in a residue extraction with transmitted light. Plane polarized, bright, or dark field illuminations all affect the angle at which the light source is reflected on to the artifact surface and is appropriate for *in situ* analysis. Cross-polarized light, in which polarizers above and below the specimen tray are activated, is a valuable characterizing tool and is only applicable when utilizing transmitted light sources.

Limitations. Limitations include artifact size, working edge angle, and depth of field issues. Larger artifacts may not have adequate working space between the optical lens and microscope stage, limiting the size of individual samples. Portable microscopes provide a possible alternative, although pilot studies currently indicate slight decreases in image quality, limitations in levels of magnification, and difficulties in accurately determining scale and magnification (Hodgson 2016b). Second, steep working edges may lack the maneuverability necessary to be positioned at a 90 degree angle from the optical lens. As a result, severe depth of field problems may be unavoidable. Prior studies effectively utilized plasticine in order to manipulate the resting angle of these artifacts. However, this process offers complications of its own should the artifacts be submitted for residue analysis at a later date. Glass or plastic mounts have been utilized in place of plasticine, but affect the

overall space available under the ocular lens, creating additional size limitations. Image stacking software circumvents the issues created through poor depth of field, but inevitably increases the time investment required for the project.

5.3 Confocal Laser Scanning Microscopy

Use-wear. Confocal laser scanning microscopy (CLSM) generates three dimensional point data that can be represented either quantitatively or through the creation of high-resolution images (Stevens et al. 2010). Surface roughness is calculable from the output data, providing characterization of edge damage and surface polish. The inclusion of quantitative research methods such as these produce increasingly detailed, evidence-based functional interpretations (Stevens et al. 2010). Additionally, the time investment required is similar to that of light microscopy, yet produces the added focal depth, magnification, and resolution more akin to that of an SEM. Magnifications possible with the instrumentation primarily range from 25x to 800x, although reports of magnifications up to 2000x can be found within the literature (Evans and Donahue 2008; Shanks et al. 2001). Due to the image capturing laser systems, casting or coating the artifacts is not required.

The mechanics of the process involve the recording of reflected light on surfaces from a specific focal plane through a pinhole aperture. The diameter of this hole determines the wavelength and depth of each focal 'slice' measured. A laser then scans the surface using a microelectromechanical resonant mirror in the laser's path. The objective lens is moved along the vertical axis and provides small packages of recorded data. Software then processes these points of light together to create a 3D representation of the scanned surface. The major benefit of the instrument is the scanner's ability to scan through the z-axis if you set the focal depth

to an area below the stage – it scans upward from the selected point until the entire surface has been scanned, creating a completed 3D surface model which can then be manipulated digitally (Evans and Donahue 2008).

Residue. CLSM has not been significantly applied to residue analysis within the existing literature. While the three-dimensional rendering may aid in the description of amorphous residues, further research is needed to determine if the resolution is adequate for fine-scaled residue observation, i.e. for starch or pollen grains.

Limitations. The technique has two inherent limitations. First, operation of the CLSM is complex in comparison to traditional light microscopic approaches. Use of the instrumentation requires training prior to unsupervised analysis. Second, the cost of the equipment is fairly expensive. While it is possible to rent blocks of time to use the equipment with research facilities or universities, the time allotments are typically priced by the hour. Depending on the scope of research being completed, this has the potential to quickly become quite costly.

5.4 Scanning Electron Microscopy

Use-wear. Scanning electron microscopy (SEM) performs two basic functions: it creates highly magnified images of specimens with little limitation in the depth of field, and it provides basic compositional data. Magnifications possible with the instrument range from 5x to 200,000x. Applications within archaeological analysis range from aiding in the identification of lithic raw material sources to identifying molecular level organic components (Frahm 2014).

The SEM emits a stream of electrons at a specimen within a vacuum. This ‘stream’ is directed by magnetic or electric fields rather than the optically controlled light source in incident or transmitted light

microscopy (del Bene 1979; Marreiras et al. 2015). The shorter wavelength of electrons allows a higher level of magnification without the distortion that results in depth of field limitations (Kooyman 2000). Different detectors record the information signals produced by the electron beam striking the specimen. The low energy of secondary electrons limits the recorded signals to those emitted within nanometers of the sample surface, providing extremely detailed images of surface topographies. Within use-wear and residue analysis, this has proven invaluable in the study of polish and striations and *in situ* amorphous residue analysis (Fedje 1979; Knutsson et al. 1986; Kooyman 2000).

Translucent lithic materials, or those with high reflective properties, are inherently difficult to adequately analyze using standard light microscopy. The use of the SEM in these conditions counteracts the need for various filters to decrease reflectivity and increase visibility of wear patterns (Knutsson et al. 1986).

Residue. Scanning electron microscopy has been utilized extensively in the analysis of starch and pollen grains (Barton 2007; Boyd et al. 2008; Haslam 2006b). Additional studies have been focused on other components of both plant and animal structures, ranging from collagen, feather, muscle tissue, bone, raphides, and multiple fiber types (Crowther 2009; Hardy and Svoboda 2009; Stemp 2001) The increase in magnification and resolution of the images provide detail not possible with optical microscopy, allowing easier identification and interpretation. Additionally, the compositional analysis feature provides preliminary inferences concerning both mineral and residue make-ups.

Limitations. Limitations of using the SEM include the physical size of the sample, the coating typically necessary prior to analysis,

investments in both time and cost, and a smaller reference collection within the literature. First, the small size of standard specimen trays limits the type of artifacts appropriate for observation. While larger trays are available for purchase, the marginal increase in size does little to alleviate the issue. Second, the conductive metal or carbon coating applied to enable or improve sample imaging is non-removable, and thus destructive to the artifact. Recent experimentation has found that it is possible to scan samples without this coating (Bouchard *n.d.*) Third, operating the instrument involves large investments in both time and cost. Access to the instrumentation is typically available at research institutions, but may incur a fee or require training in order to operate. Due to the increased scale of magnification, the analysis of complete artifacts is extremely time consuming, further increasing the cost and duration of the project. Taking a sampling approach and limiting observations to pre-determined portions of the artifact helps mitigate the cost. Lastly, there is currently a limited experimental reference collection within the literature; perhaps due to the aforementioned issues. While this collection has grown significantly in recent years, it does not yet compare in size to those available for other microscopic approaches. This will continue to change as the method develops and technological advances are made.

5.5 Biochemical Testing

Residue. Biochemical testing determines the presence or absence of specific classes of compounds within a residue mixture through pre-established colorimetric responses. While the results differ, the basic mechanism behind the process remains the same for each individualized test. The practice is currently used in several fields of research, including forensics, biochemistry, and biomedicine (Cook 2015; Matheson and Veall 2014). A multitude of these tests exist within the

current literature and can detect targeted compounds including, but not limited to, carbohydrates, starches, fatty acids, proteins, nucleic acids, and alkaloids (Benedict 1909; Bradford 1976; Briuer 1976; Soloni and Sardina 1973).

Limitations. Despite the rapid development of the approach, two important limitations need to be addressed when selecting a sample. First, the minimum concentration threshold to indicate the presence of a compound using these tests is unknown. This limitation is particularly relevant as most archaeological residues are in small quantity, inherently unknown in composition, and likely represent mixtures. Second, the tests are limited to identifying the presence of the compounds; they cannot determine the relative age, authenticity, or source of the compound. Post-depositional and modern contaminants can also react positively to the test. For this reason, the use of biochemical testing should always be used in conjunction with multiple analytical techniques.

5.6 Absorbent Light Spectroscopy

Residue. Absorbent light spectroscopy measures differential light absorption over varying wavelengths by functional groups at a molecular level (Matheson and Veall 2014). The instrumentation may be used in two ways. First, the instrument can produce spectrographs illustrating the differential absorbance rates, or peaks, of molecules within the residue. Successful identification of lipids, fatty acids, metals, and nucleic acids have been completed in previous studies (Malainey 2011; Price and Burton 2010). Second, if biochemical test protocols have been optimized for immediate use with the spectrometer, the instrument can be used to quantify the data numerically. The numerical data emitted from archaeological samples can then be compared to those of sample blanks run prior to determine relative baselines for positive or negative test results.

This is particularly relevant due to the undetermined minimum thresholds in biochemical testing; numerical data may indicate a positive result in cases where colorimetric positives were not observed.

Limitations. There are not significant limitations to this approach when used to quantify the relative concentrations of biomolecules within the sample. The equipment is usually available at academic or research institutions and may require a small fee to use, and requires minimal training. If employed in an identifying capacity, caution is strongly recommended with interpretation; additional lines of evidence should always be pursued. Comparative data exists within the literature, but is not exhaustive and may require additional experimentation by the researcher.

5.7 Gas Chromatography coupled Mass Spectroscopy

Residue. Gas chromatography coupled mass spectroscopy (GC-MS) is an analytical technique that has been developed in the field of chemistry and is now being applied to a broader range of disciplines. The technique is based on the separation of molecules as they travel in a gaseous phase through the gas chromatography (GC) column (Malainey 2011). Gas chromatography has the ability to separate the individual constituents in complex organic mixtures (Malainey 2011). This is followed by a structural characterization of the purified compounds by a mass spectrometer (Brown and Brown 2011; Malainey 2011). The technique has successfully characterized waxes, resins, alkaloids, amino acids, carbohydrates, and hydrocarbons from archaeological materials in previous studies (Columbini et al. 2005; d'Errico et al. 2012; Evershed et al. 1997b; Evershed 2008). The generated data can then be compared to known samples, allowing for a positive identification. Gas chromatography coupled mass-spectroscopy

is an effective method due to its ability to separate and characterize mixtures and identify sources of contamination from both environmental and anthropogenic sources (Evershed 2008; Veall and Matheson 2014).

Polar compounds or compounds with polar-functional groups that contain oxygen or nitrogen require derivitization prior to GC-MS analysis. Freeze drying the samples prior to derivitization ensures the purity of the solution and limits interference caused by possible contaminants (Cook 2015; Orsini et al. 2015). If the residue does not contain polarized compounds, or has been removed using a non-polar solvent (i.e. hexane), derivitization is not necessary. Preparation is highly dependent on the extraction solvents used, and the appropriate literature should always be consulted prior to the study.

Limitations. The time and cost required for sample preparation, testing, and analyzing may limit the sample size possible. While academic institutions often have discounted pricing for in-house research, a large sample size can quickly increase the cost of research. Additionally, the time required to analyze the results is highly dependent on the researcher's familiarity with chemical analysis, and may become very time consuming for the novice.

The minute amount of residue typically recovered through residue extraction increases the chance of contamination and dilutes the archaeological compounds within the mixture, potentially creating difficulties in the mass spectroscopic analysis. Steps to avoid this include stringent observations of lab protocols, and the testing of a blank sample in order to determine any background static present (Malainey 2011).

6.0 Data Analysis

The methodologies discussed thus far have established analytical and interpretive

protocols. If the reader intends to pursue or practice any methodological approach discussed here, it is highly recommended that they complete a review of the existing literature. The overview of terminology provided in the following pages provides a basis on which a novice analyst may need to begin a multi-analytical study of lithic artifacts. Use-wear and residue analyses are discussed separately, with appropriate data collection instrumentation discussed as needed. The terminology required for both broad techniques essentially remains similar independent of the scale of instrumentation employed.

6.1 Use-wear

Use-wear analysis refers to the study of wears, or damages, located on the edge or surface of artifacts that result from utilization (Fullagar and Matheson 2013; Odell 2003). The term *microwear* is sometimes used interchangeably with use-wear, but may be used in reference specifically to the high-powered microscopic analysis of polishes. The term *traceology* is likewise used interchangeably with microwear, but refers to the study of all traces of wear, including both residue and use-wear (Fullagar and Matheson 2013). The fracture mechanics involved in the determination of morphological scar properties (initiation, termination, orientation, size) are determined by force application, edge morphology, and the hardness or resistance of the material worked. The latter is sometimes referred to as the source or contact material. Several approaches to analysis exist, all of which focus on aspects of microchipping and features including polish and striations, amongst others. Microchipping focuses on the morphology and distribution of flake scars resulting from use while feature

analysis concentrates on the broader, more amorphous signs of wear. Feature analysis frequently focuses on the formation and type of polish and striations, but also includes additional features such as nibbling, crushing, rounding, smoothing, crazing, stepping, and snap fractures (Keeley 1980; Odell 2003; Tringham 1974). Each of these features will be discussed below, and are visible to varying extents in each of the microscopic approaches.

Microscopic flakes and flake scars share the attributes and characteristics of their macroscopic counterparts. The resulting shapes appear scalar, trapezoidal, triangular, rectangular, or as a crescent (or half-moon) (Fig. 1), and are influenced by a multitude of variables. These include source material hardness, resistance or elasticity, manner of use, and lithic material variability. The initiation scar is located on the proximal end of the scar and can range from nearly flat to curved in appearance. Flake termination scars indicate the distal end morphology of the micro-fractures and are most frequently feathered, stepped (type 1 or 2), or hinged¹ (Fig. 2). Feathered terminations gradually become shallower until they meet the non-damaged surface, while stepped terminations end abruptly in a right angle break. Step type 1 displays a clean break, while the type 2 variety displays a ‘cover’ of extremely thin lithic material due to an incomplete flake detachment. Hinge terminations ‘roll’ out to

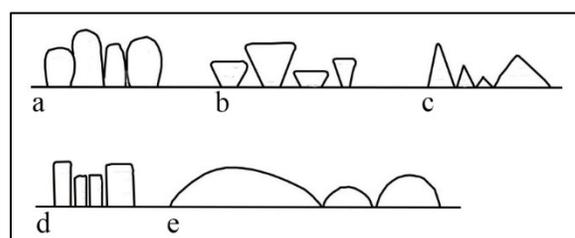


Figure 1: Morphology: scalar (A), trapezoidal (B), triangular (C), rectangular (D), halfmoon (E).

¹ May be referred to as alternate terminology: regular, reflected, stepped, oblique (Marreiros et al. 2015)

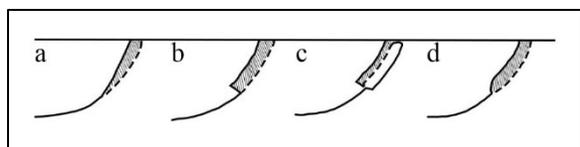


Figure 2. Termination types – feather (A), step type 1 (B), step type 2 (C), and hinge (D). (Modified from Cotterall and Kamminga 1987).

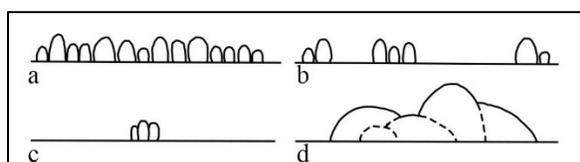


Figure 3. Distribution variations – continuous (A), discontinuous (B), isolated (C), and superimposed (D).

the dorsal surface, resulting in a rounded or curved distal edge (Kooyman 2000). The distribution of edge damage can be referred to as continuous or discontinuous, as well as isolated, aligned, or superimposed (Fig. 3).

While microchipping is primarily limited to edges of the lithic, excluding hafting or post depositional wear, features are found on both the working edge and surface area of artifacts as a result of gripping or hafting modifications. Polish has been extensively studied, and has proven to be a diagnostic indicator of source material (Kamminga 1979; Keeley 1980; Gibaja and Gassin 2015; Rots 2010; Van Gijn 2010). While visible with low-powered microscopy, it becomes increasingly diagnostic at higher magnifications. The formation of polish can be either additive or destructive in nature. Additive polish is the result of a buildup of silica rich materials, in which material is gradually added to the tool edge, creating a superimposed gloss on the lithic material.

Destructive polish is the result of a gradual but steady wearing down of the mineral matrix along the working edge. As wear develops and microflakes are detached, they become part of the abrasive make-up which also includes environmental (e.g. dust, sand) and use-related abrasives (bone, fur, fats, etc). This abrasive material is rubbed between the source material and the remaining stone tool edge, resulting in the tribochemical breakdown of the edge, forming an altered mineral surface (polish) over time (Dubreuil and Savage 2014).

The polish can be continuous or discontinuous depending on the source material and lithic material variability, and can have bright or dull spots within the homogenous mass. It can also appear patchy, glossy, greasy, dull, bright, or resinous (Fig. 4). Striations are a secondary result of the abrasive mixture, and can also vary morphologically, again dependent on the lithic and source materials involved. They are sometimes difficult to observe at lower magnifications, but at higher magnifications are distinguishable as parallel or perpendicular to the working edge. Observation with CLSM or SEM results in discernable striation depths and textures, providing information concerning striation formation and possible source. Regardless of the scale at which striations are observed, they provide information concerning the direction of tool motion and causal function.

While features may lack the diagnostic capabilities of polish and striations, their cumulative documentation aids in the overall determination of tool function (Tringham et al. 1974). Nibbling and crushing are both the result of forceful

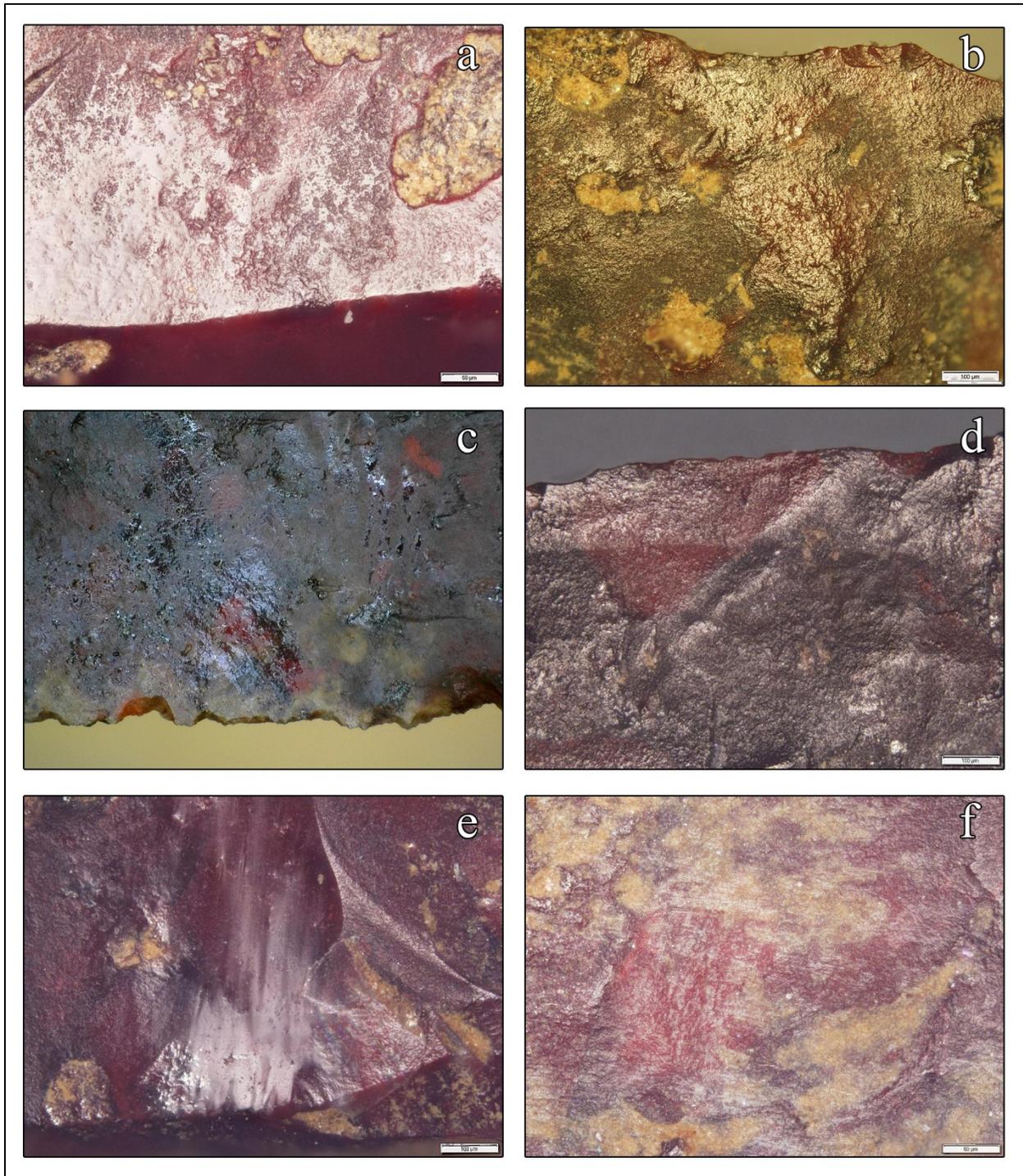


Figure 4. Examples of polish types including bright (A), patchy (B), greasy (C), dull (D), resinous (E), and with striations (F).

application to medium or hard material types (Fig. 5). They can both be continuous across working edges, or limited to discrete locations with small areas of thinner or more fracture-prone material. While crushing tends to result from forceful contact with harder materials, nibbling will occur from softer materials with higher levels of elasticity, or resistance. Rounding and smoothing occur in the early stages of polish formation, but can be observed without the presence of a noticeable polish (Fig. 6). Stepping is an accumulation of stepped or hinged fractures superimposed on one another, and is the result of repeated use on medium to hard materials. The appearance of this type of damage looks similar to repeated retouch, and can typically be distinguished based on scale, location, and distribution (Fig. 7). Snap fractures are the result of use on a material of any hardness with high levels of elasticity, or are due to natural fault lines within the mineral matrix of the lithic material along the working edge. Macromorphic snap fractures are particularly common in hafted tools from the differential force applications caused by higher stress levels placed along the mid-section of the tool (Lozny 2004). These variables are affected by a multitude of options, primarily due to the manner of use (e.g. direction, force, grip, impact type), or the source material (e.g. hardness, elasticity, lubrication), and can be the result of post-depositional damage.

6.2 Residue

Locard's exchange principle states that an exchange of material will take place whenever two or more materials come into contact; this is often cited as the keystone principle of residue analysis (Briuer 1976; Haslam 2006; Hortola 2005; Loy 1993). Residues refer to any material that has been transferred and remained adhering to artifacts through direct or indirect use, e.g. cutting wood, or being hafted in a wooden haft

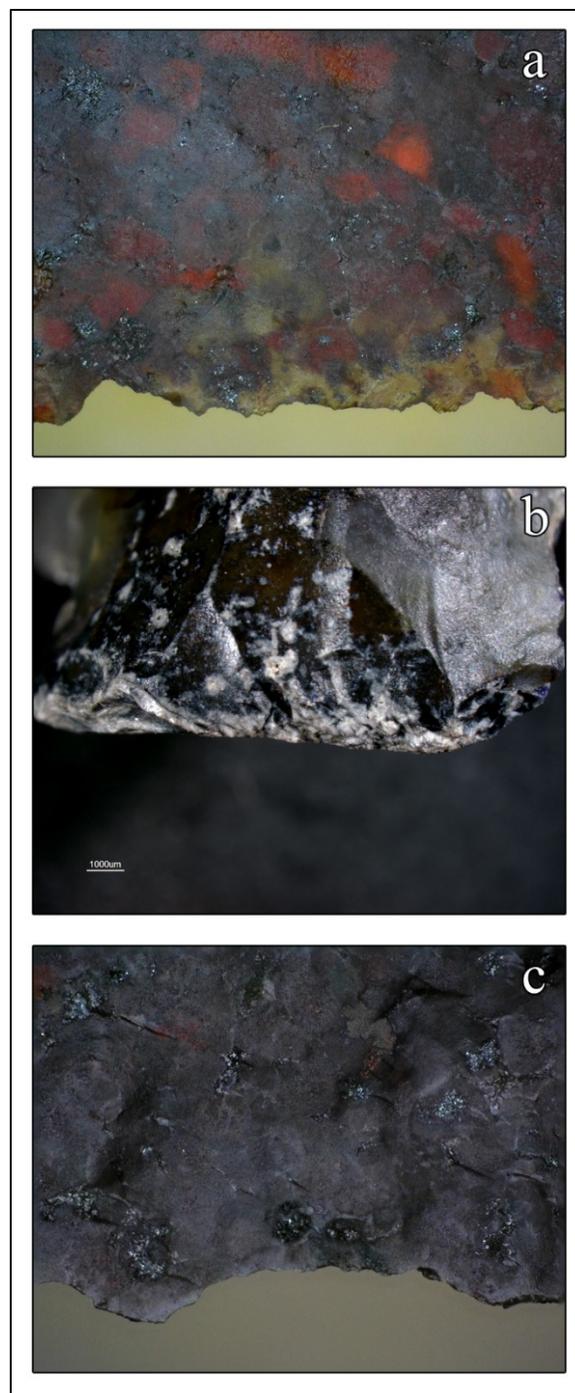


Figure 5: Examples of nibbling (A), crushing (B), and both combined (C).

(Fullagar and Matheson 2013). Residues are characterized through the identification of diagnostic microfossils, compound structures, chemical signatures, atomic

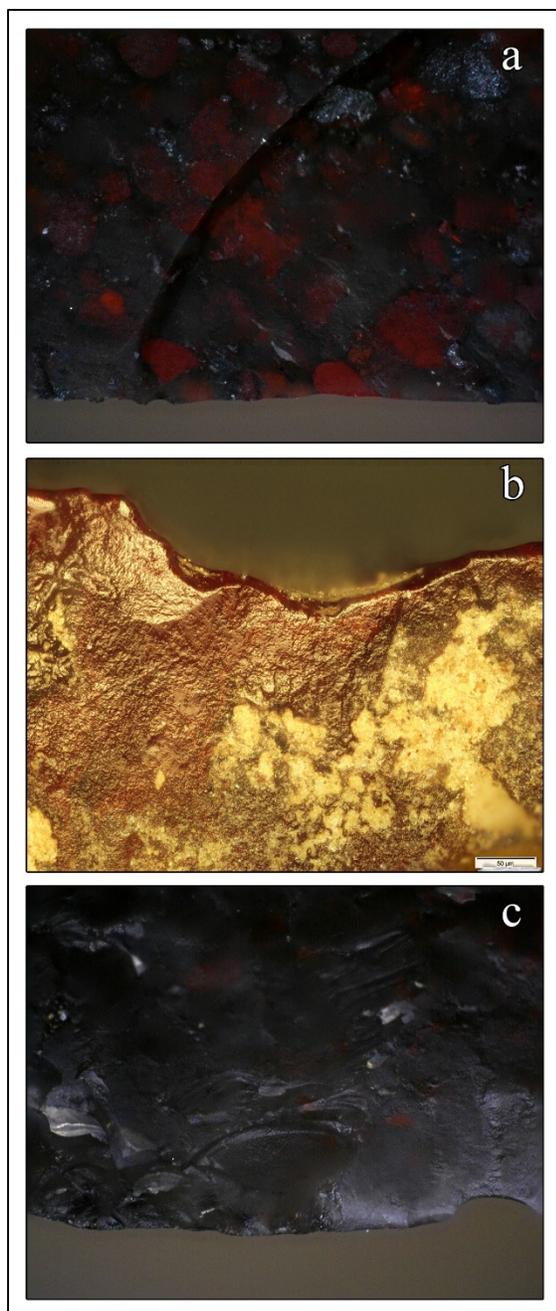


Figure 6. *Smoothing and rounding. Extensive smoothing (A), minor rounding with polish (B), and a combination of smoothing and rounding (C).*

structures, and genetic composition (Fullagar and Matherson 2013; Odell 2003). Examples of microscopically visible compounds include, but are not limited to, plant components (starches, phytoliths,

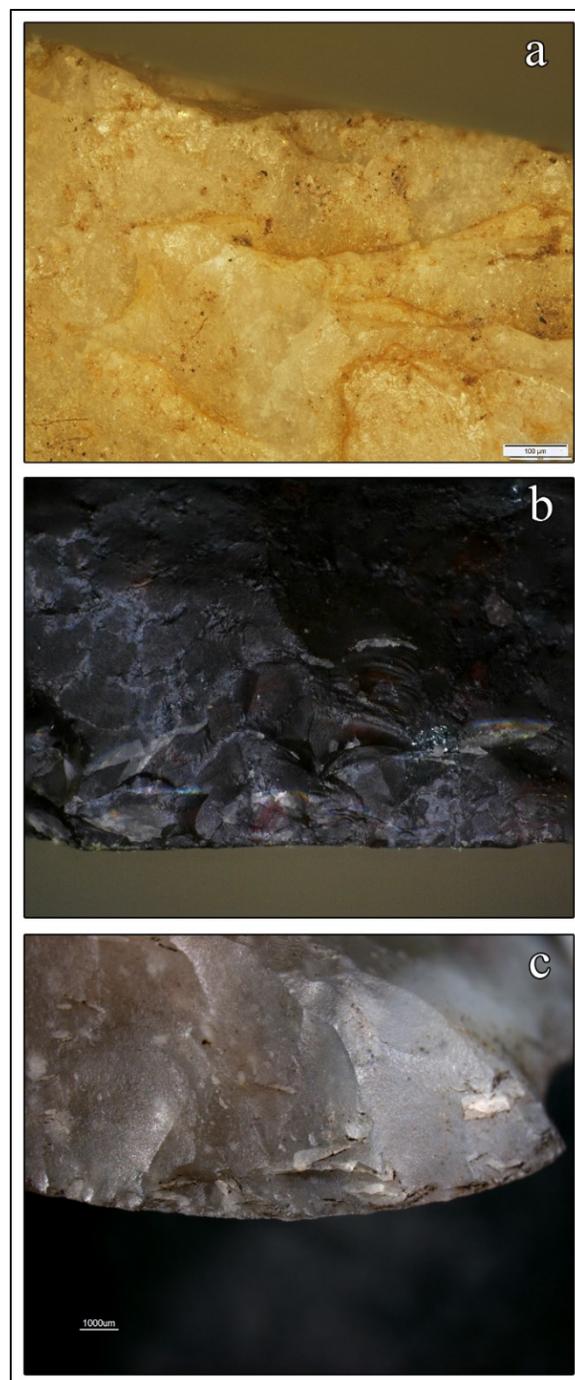


Figure 7. *Examples of stepping and retouch on chert (A), taconite (B), and chalcedony (C).*

pollen grains, schlerieds, raphides, tracheids, druzes, raphides, etc), keratin structures (hair, scale, feather), plant or animal fibers (cellulose, collagen, hair), insect fibers or parts (chiton), synthetic fibers (historical or contamination), lithic materials (flakes or

pigments), amorphous residues (blood, honey, pitch), charcoal, or biomolecules. Chemical residue analysis is targeted at archaeological biomolecules, specifically their organic chemical compounds.

Microscopically visible cellular components are referred to as compound or morphological structures. While larger structures are sometimes visible with magnification under 100x, higher magnifications produce the resolution necessary to adequately characterize and describe structures of all sizes. Adequate description increases the accuracy of subsequent identification and interpretation of the residue. Important descriptors include the size, form, birefringence, luster, translucency, and response to cross-polarized light (Langejans and Lombard 2015). Pertinent descriptors will be discussed below.

Plant components are found within all stages of microscopic analysis. Starches, phytoliths, and pollen grains have been extensively studied and documented (Barton 2007; Boyd et al. 2008; Haslam 2006b). Size, morphology, and birefringence are used to determine the source of unknown microfossils, and can be diagnostic to genus or species when compared with known samples. Size typically varies from 1 μ m to 175 μ m, and the shapes, sizes, lamella, position of hilum, and polarization can be specific to different plant species (Langejans 2006; Langejans and Lombard 2015). Extinction crosses in starches are particularly identifiable and are a frequent indicator of starch type. Additional plant components consist of vessel or structural elements including sclereids, raphides, tracheids, and druzes, amongst others. Size in the form of these structures can vary widely (Fig. 8).

Each produces extinction colors when exposed to cross-polarized light, typically emitting a blue to white glow. Extinction crosses are not present in non-starch cells. In cases where cellular tissue has been observed

as opposed to individual cellular components, an identification of 'plant material' can be made. Plant tissue is bright and anisotropic under cross-polarized light (Langejans and Lombard 2015:204). Degraded plant tissue is frequently warped, fragmented, or leached of color, and will not have visible chlorophyll. If charred, it may appear darker or increasingly opaque under plane polarized light and will not emit light under cross-polarized observation (Fig. 9).

Keratin is the sulfur-containing fibrous protein that forms the basis of epidermal tissues including hair, scales, horns, and feathers (Chernova and Kirillova 2010; Langejans and Lombard 2015; Robertson 2002). While modern keratinous materials are fairly easy to identify, archaeological samples may be highly degraded and consist only of very small fragments which may be altered in appearance. Each of these structures has a distinct form under microscopic observation (Fig. 10). Depending on which microscopic attributes have been preserved, the structures may be identifiable to genus or species. A wide range of comparative data exists within the current literature.

Natural fibers fall in to one of four categories: plant, animal, insect, or environmental (Petrao and Kubic 2004) (Fig. 11). Synthetic fibers may be observed as well, and are typically the result of contamination. Common plant fibers include kapok, cotton, flax, hemp, jute, ramie, abaca, sisal, and wood. Plant fibers are typically translucent under plane polarized light and bright and anisotropic under cross-polarized light. They are cellulosic and consist of elongated, narrow cells that resemble flat and twisted strands of ribbon with shattered ends (Langejans and Lombard 2015). Cell structure, shape, size, cross-markings, crystal shapes, lumen size, length and width are used to identify and categorize the fibers. Wood fibers can be differentiated based on the

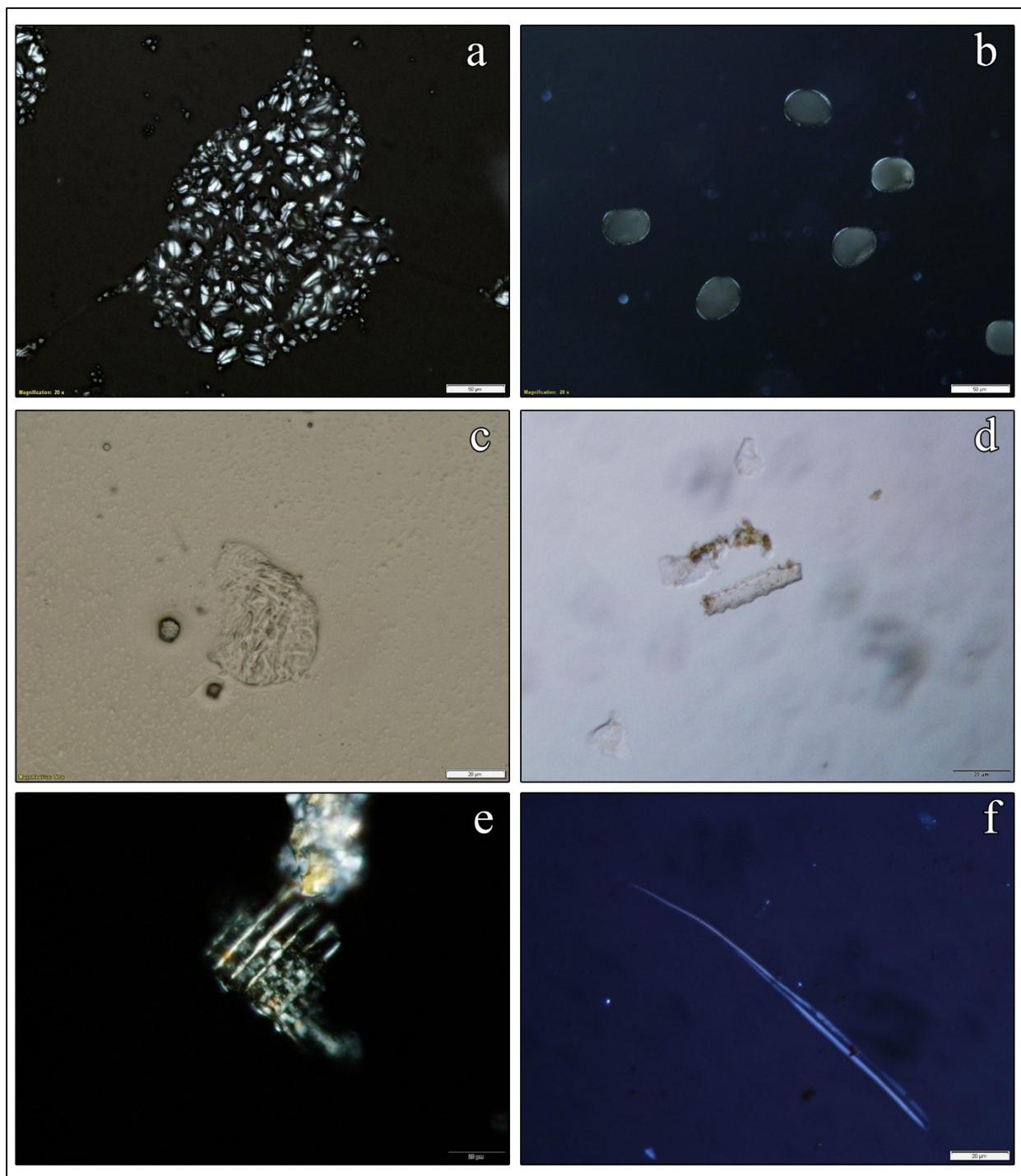


Figure 8. *Microscopic plant components – potato starch (A), corn pollen (B), squash phytolith (C), grass phytolith (D), wood (E), and a raphide (F).*

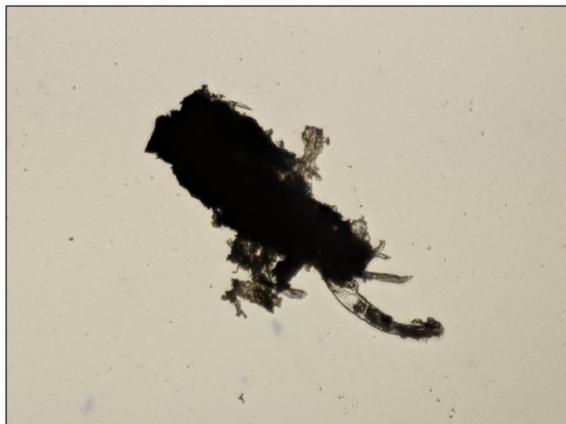


Figure 9. *Carbonized plant tissue.*

presence of cross-field pitting, ray tracheids, spiral thickenings, vessel elements, and ray pittings (Petraco and Kubic 2004). Common animal fibers include collagen and hair (or fur). Collagen fibers can be observed individually or in bundles of 2-10mm in diameter. Low collagen counts result in a loose weave (i.e. skin and muscle connective tissues), while higher amounts of collagen result in much denser organization (i.e. tendons or fibrous connective tissue). While colorless and nearly opaque under plane polarized light, these fibers appear whitish-blue under cross-polarized light. In degraded specimens, the terminations of collagen bundles can look similar to unravelling rope. Hair and fur appear cylindrical and consist of three layers: cuticle, cortex, and medulla, the outer layer, main body, and central canal respectively (Lombard and Langejans 2015; Petraco and Kubic 2004). The cuticle is composed of overlapping scales which create imbricate, mosaic, petaloid, or chevron shaped scale patterns. When combined with the medullary index (the ratio of the hair shaft and medullary diameters), species or genus interpretation may be possible. Silk is the primary insect fiber observed in archaeological analysis, and can be identified by the same criteria as the natural plant fibers, or by using dispersion staining techniques (Petraco and Kubic 2004). Environmental

fibers are those that occur naturally within the burial environment. The most frequently observed of these are hyphae, a structural component of fungi. The fibers are made of several cells, and can resemble a strand of hair to a novice analyst.

Amorphous residues lack definitive form, and indicate substances that had existed in a liquid or semi-liquid form prior to adhering to the artifact surface. Due to their lack of diagnostic characteristics, these residues can be difficult to identify using microscopic techniques alone. *In situ* characterizations are based on color, texture, and luster (Bouchard *n.d.*). Further identification requires extraction, at which time cross-polarized light microscopy, enzymatic digestion, and the use of dyes in addition to chemical and spectroscopic analysis can be applied. Amorphous residues vary widely; examples include fatty tissues, plant exudates, resin, or gum; dried fluids like milk, beer, egg, or blood; and any number of adhesive mixtures (Fig. 12).

Inorganic structures are identifiable within residue extractions and can consist of lithic or metal materials, the latter of which will not be discussed here. Although uncommon, use-related microflakes can be observed within residue extractions (Hodgson 2016a). Interpretation of these artifacts should be taken with caution, as microscopic sediments from the burial environment may be included within the observable residue. Identification of these flakes is similar to that of their macroscopic counterparts; ideal specimens will contain bulbs of percussion, platforms, and distinguishable terminations. Less commonly, striations or other indications of use may also be visible (Fig. 13). Pigment, a second visible lithic type, has been documented more extensively than microflakes. Iron oxides of various colors were used as dyes or as part of binding recipes in certain regions (Lombard 2006).

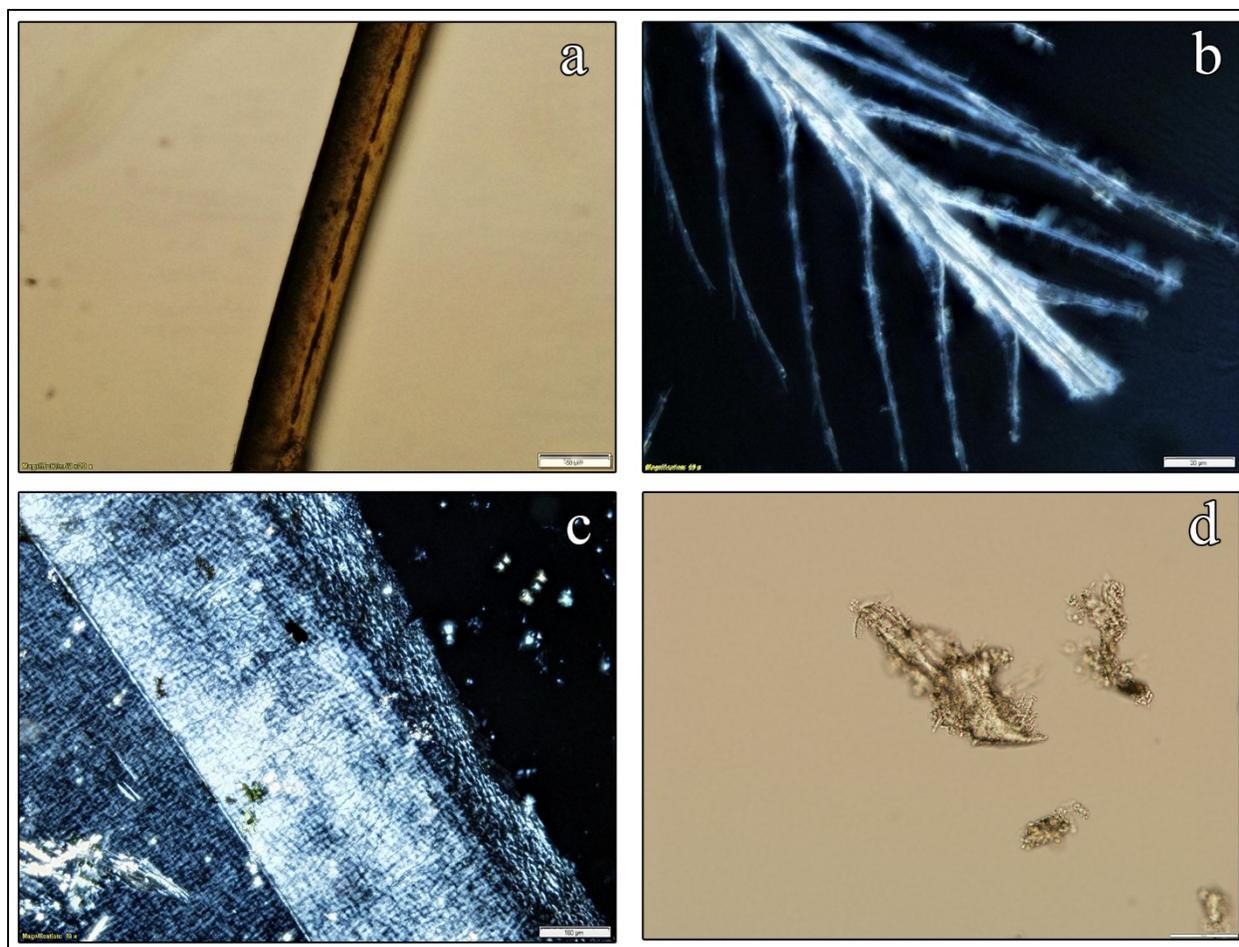


Figure 10. Keratinous structures including hair (A), feather (B), scales (C), and horn (D).

They are visible at all stages of microscopy, and will appear as their natural color (i.e. yellow, red, purple) under plane polarized light and lack defined boundaries; birefringence is dependent on the specific molecular make-up of pigment and typically results in a dull-glow of the natural color. As an inorganic substance, they are not detectable through organic chemical analysis, but can be determined through infrared microspectroscopy, energy dispersive x-ray analysis, or Raman spectroscopy (d'Errico et al. 2012; Petraco and Kubic 2004).

Archaeological biomolecules are the large organic compounds of once living organisms that are sometimes preserved in various states of degradation within residues

(Brown and Brown 2011). These macromolecules fall into four broad categories: nucleic acids, proteins, lipids, and carbohydrates. Nucleic acids (DNA and RNA) are not typically observable without specialized methodological approaches and instrumentation. They also require stringent lab protocols. For additional information on nucleic acids, please consult Brown and Brown (2011) and Malainey (2011). Proteins are found in all structural and functional roles and include bones, teeth, hair, and structural cell components such as collagen or blood. They are observable with both optical and SEM approaches, and are detectable through chemical analyses. Lipids are a highly diverse group that includes fatty acids, fats

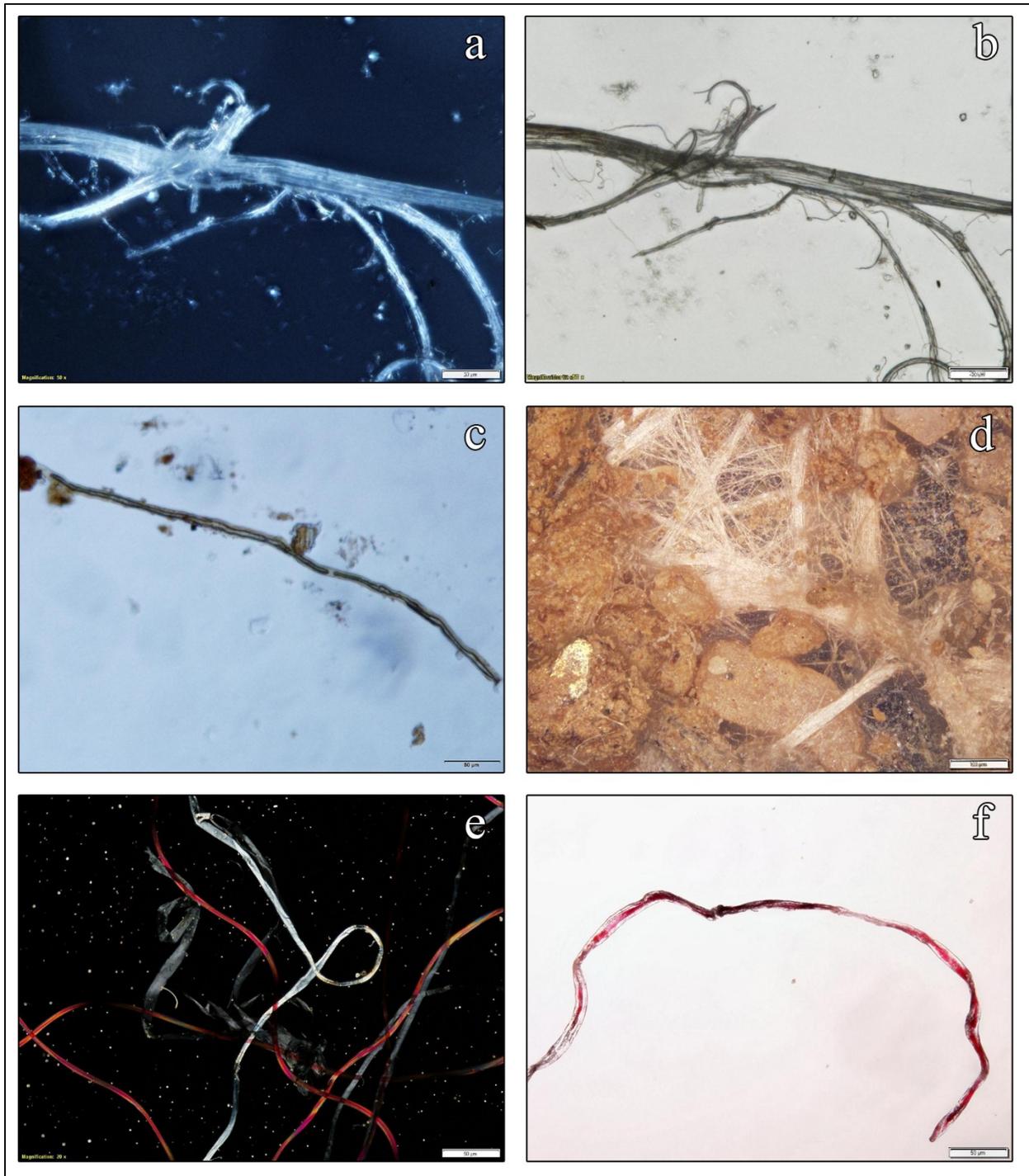


Figure 11. Various fibers – animal sinew (A, B), natural fibers (C, D), and synthetic fibers (E, F).

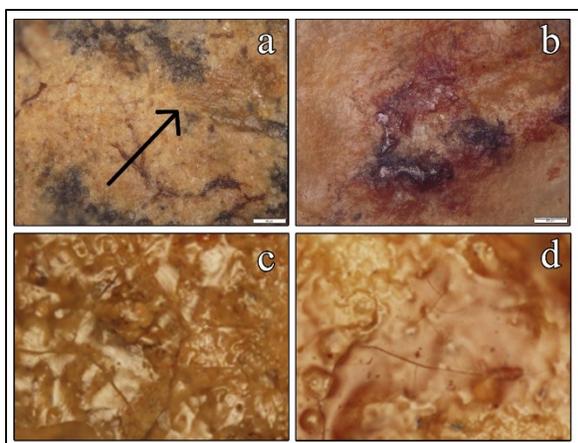


Figure 12. Amorphous residues including striated muscle tissue (indicated by arrow, A), pitch (B), egg (C), and blood (D). Egg and blood images modified from Matheson 2013).

and oils, waxes, steroids, and terpenes. They have both structural and functional roles and are typically fairly well preserved due to their hydrophobic nature. Because of their variety and specialized functions, they can be used to determine specific materials, i.e. the tree species of a pitch used within an adhesive mixture. Lastly, carbohydrates are both structural and storage compounds and take the form of starch and cellulose in plants, or glycogen in animals. Starch and cellulose are both visible at all stages of microscopy, and all three can be detected through chemical analysis.

The original biological structure of ancient biomolecules is rarely intact and almost always modified. Rather, the observation is of the preserved version of that structure; a structure which has likely undergone modifications prior to burial (e.g. cooking) or within the burial environment; for example, acidification and natural breakdown processes (Brown and Brown 2011). Specific burial environments should be considered prior to an analysis to familiarize the analyst with possible modifications. Post-excavation storage may likewise cause modification to organic remains; modern contaminants, storage,

curation or preservative activities may introduce variables needed to begin or expedite degradative processes.

7.0 Data Interpretation

Interpretation is the phase in which meaning, or function, is determined based on the cumulative qualities of all observed variables. Use-wear analysis contributes to the determination of the manner of use and begins to narrow the possibilities of source material. Residue analysis verifies and elaborates on the preliminary findings of use-wear analysis, and may provide specificity to genus or species level of any present residue. Each provides multiple independent lines of evidence that together create a strong final interpretation.

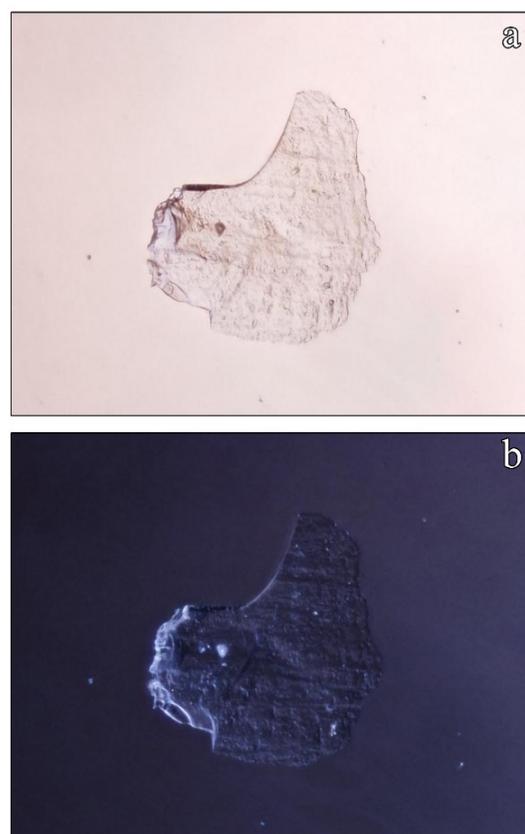


Figure 13. Chert use-flake found within a residue extraction with striation and edge damages visible.

7.1 Use-wear

The function of an artifact is interpreted through an analysis of contact material hardness, manner of use, and style of grip. The hardness of the contact material affects the formation of micro-wear traces throughout use, while the manner of use (e.g. direction, force) affects the location and type of damages that are possible throughout the completion of a task. The manner of grip, hafted or handheld, likewise affects the location of the resultant wear patterns and the amount of force applied throughout use. Each factor is interpreted through a combined analysis of the distribution, extent, and morphological variability of all observed attributes. The next several paragraphs discuss common wear patterns observed in each category.

Source Material Hardness. Source material characteristics are determined by micro-scar morphology, termination, distribution, and micro-feature formation. The relative hardness of the source material can be determined through low-powered microscopic analysis. Increasingly detailed analysis is only possible with greater magnification. The formation of wear patterns, though distinguishable at later stages of use, may appear similar in 'lightly used' artifacts; scars of each morphological type may be observable on the working edge at this stage, regardless of the contact material hardness. The ratio and relative frequencies of these scars, rather than their presence or lack thereof is the determining factor in the interpretation of source material (Tringham 1974).

Softer materials include meat, plant materials (roots, tubers, etc), and hide. The softness of these materials provides more intrusive contact with the tool edge, decreasing the force applied and limiting the subsequent fracturing of the edge (Lawn and Marshall 1979). This results in relatively few

flake scars when compared with tools used on harder materials, with feathered scalar scars being the most common. The increased contact between material and tool also results in the formation of intrusive polish which forms continuously across the contact edge, causing both smoothing and rounding over time. With higher magnifications, characterizations of polish can become significantly more detailed. For example, fresh hide polish is described as 'greasy' and bright due to the large amount of lubrication (animal fat) deriving from use, while dry hide polish is characterized by heavy rounding and smoothing with dull, pitted, and matte polish with striations perpendicular to the tool edge (Loebel 2013). Plant polish is described as glossy or liquid-like, and is the result of additive polish with a high volume of silica particles. Striations may be visible due to micro-flakes or other abrasive materials getting trapped between the contact material and working edge, aiding in the identification of manner of use (Brink 1978). Crushing and stepping are very uncommon from use on soft materials, although stepping as a result of failed retouch may be visible. Macromorphic snap fractures, where the working edge of a scraper has snapped off, are common with hafted scraping tools, but are less visible microscopically on the working edges themselves.

A broad range of medium materials exist, including fresh and seasoned wood, woody plants, and fresh bone (Tringham 1974). The resistant yet pliable nature of these materials results in more frequent edge damage, typically in the form of trapezoidal and triangular scars with hinged or stepped terminations. Due to the broad nature of this material type, the formation of polishes and other features is more variable than those observed in soft material types. For example, fresh wood or bone maintain the elasticity required to permit more invasive polish formation, while their seasoned alternatives

do not. Wood plant material may form polish in much the same way as softer plant materials like tubers, yet have wear patterns indicative of harder materials. The distribution varies according to the specific hardness and elasticity of the material: it can range from continuous to patchy across the working edge, and can develop isolated bright spots. The invasiveness of polish is also variable: it may be limited to the outermost edge, or it may intrude several millimeters onto the surface; the invasive polish may be patchy, or heavy, or have distinct areas of each. In comparison to polish from softer materials it is almost never glossy. Even when continuous, duller patchy areas are common in addition to the aforementioned bright spots.

Harder materials include fresh and seasoned bone, seasoned wood, and antler. Materials such as these tend to leave a greater number of triangular scars intermixed with scalar, although trapezoidal and rectangular scars are not uncommon. Stepped and hinged terminations frequently occur, with very few if any feathered terminations. The accrual of wear in discrete clusters across the edge is common due to the harder nature of the material. With repeated use, these clusters eventually spread in a continuous manner across the working edge. Despite this, it is the cluster stage which is most frequently visible within use-wear analysis. This is because the accumulation of heavy damages such as these almost always results in the re-sharpening of a tool edge and subsequent obliteration of all previous wear. If the edge has been exhausted, it is usually discarded shortly after the final re-sharpening (Loebel 2013). The polish of harder materials is typically located only within near proximity to the working edge. Heavy use results in fairly continuous polish across the working edge, although it tends to be more developed on sections of elevated micro-topography. Crushing is more likely to result from hard material use, as are

stepping and snap fractures. Rounding may occur, but is limited only to the outermost angle of the tool edge. Higher magnification is typically needed to accurately differentiate between polishes from medium and hard materials.

Manner of use. The manner of use encompasses longitudinal, transverse, or circular motions, unidirectional or bidirectional movements, and obtuse to acute functional angles (Fig. 14). Longitudinal and transverse motions respectively describe movements which travel lengthwise or cut across the material, while circular motions depict clockwise and counter-clockwise movement of a tool tip at <90 degrees angle to the contact material. Unidirectional movements imply a single-direction propelled movement (push or pull), while bidirectional movements indicate the use of both together (push and pull). Functional angles depict the angle at which an artifact was used, typically spanning acute to perpendicular angles; the angle or angles of use affect the severity and distribution of subsequent edge damages. Each of these attributes are determined by the location and relative amount of wear on both aspects of the tool (dorsal and ventral), and the orientation of microchipping or striations (right or left oblique, perpendicular or parallel to the working edge).

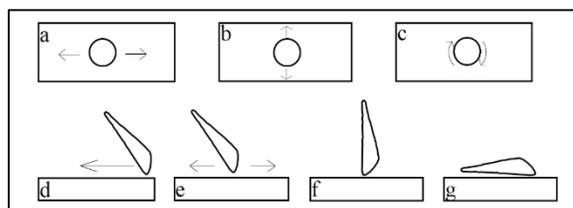


Fig. 14. Demonstrations of variables effected by manner of use – longitudinal (A), transverse (B), circular (C), unidirectional (D), bidirectional (E), 90° angle (F), acute angle (G).

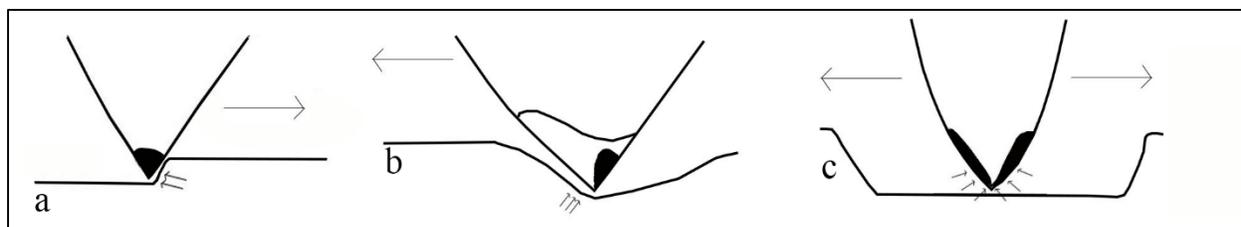


Figure 15. Variations of planing (A), scraping (B), and cutting (C). Direction of use and force are indicated by large and small arrows, respectively. Borders near tip indicate extent of polish, and shaded areas depict microwear zones.

The relative ratios of wear on either side of the tool are indicative of the movement of the tool. Microchipping damage limited to one side of the working edge is indicative of unidirectional tool use. Equal amounts of damage on either side indicate bidirectional movement, in which a tool was used equally in more than one direction. If damage is present on both sides but more predominant on one, transient bidirectional use (or incidental use) is possible.

To illustrate the differences, consider the following scenarios (Fig. 15). A steep angled working edge displays microchipping and striations perpendicular to the dorsal edge. These damages indicate a pulling motion in which the ventral surface contacted the material first, initiating a fracture and resulting in the removal of flakes from the dorsal surface, such as in some scraping or planing motions. In an alternate scenario, the same damages may be located on the ventral surface in addition to extensive polish and striations situated perpendicular to the edge. This would indicate initial contact with the dorsal surface in a pushing motion on a much different source material, such as scraping a stretched hide while being hafted in an L-shaped haft. Last, consider a tool edge with equal proportions of wear on both the ventral and dorsal surfaces in the form of stepping, nibbling, and a high frequency of hinged triangular and scalar scars. This would indicate bidirectional cutting movements on a harder material such as bone. These

functions as well as sawing, drilling, whittling, and others are discernable through use-wear analysis. Functional analyses via use-wear analysis are also possible for groundstone artifacts; however, this type of functional analysis is beyond the scope of this paper. See Dubreuil and Savage (2014) for more detailed information.

Style of grip. The style of grip refers to how an artifact was held during use; whether it was hafted or handheld. If it was hafted, which style was used? The grip of an artifact affects the maneuverability; this includes the angle of use, the leverage possible, and the amount of force employed. Identifying the presence or absence of hafting within a site assemblage affects not only site interpretation, but more general theories of technological evolution and human behavioral capacities (Ambrose 2010; Rots et al. 2015). Extensive studies have been completed by Rots to determine the differences between handheld and hafted grips (Rots 2005, 2006 2010). Hafted grips can be further subdivided into male, male split, juxtaposed, or wrapped hafting styles (Fig. 16).

Male hafting arrangements involve the insertion of the stone tool into the haft, while juxtaposed arrangements see the tool being placed on top of the hafting material. Male split hafting involves splitting the haft prior to tool insertion. In all three cases the tool is bound in place with binding, resin, or a mixture of the two. The hafts are typically

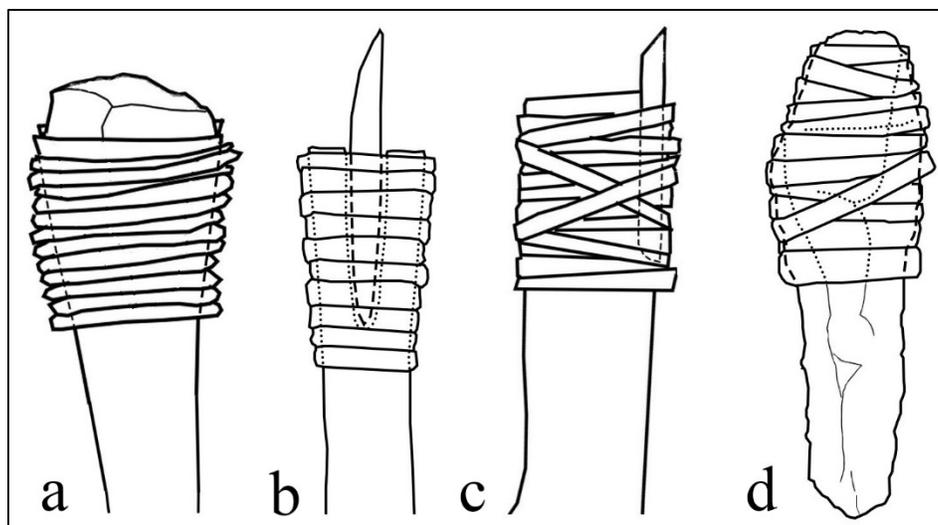


Fig. 16. *Hafting styles – male (A), male split (B), juxtaposed (C), and wrapped (D).*

made of wood, bone, or antler. Wrapped hafting uses leather, wet leather, intestines, or vegetal material applied directly to the artifact itself to form a grip.

Attributes used in the determination of hafting include polish, scarring, and bright spots. Hafting polish is restricted in distribution and has a well-defined zone. Scars are frequently scalar and trapezoidal with hinged terminations, with possible crushing and scar overlapping. Bright spots are frequent and typically large in scale. The analysis of increasingly detailed attributes of polish and scarring allows differentiation between hafting or binding styles and materials. For additional information, please consult Rots (2010).

7.2 Residues

While use-wear analysis is primarily concerned with the determination of tool function, residue analysis focuses on a more detailed interpretation of source material. Interpretation is inherent to each stage of residue analysis, from the identification of microscopic structures to the interpretation of spectrographic data attained through chemical analysis. Despite this variability, the broader interpretive process remains the same

The interpretive process of organic residues involves six tiered questions (Matheson and Veall 2014) (see Chart 1). First and foremost, the presence or absence of residue must be determined; macroscopic or low-powered analysis is typically adequate. Second, the organic nature of the residue needs to be determined. This is accomplished through the identification and analysis of organic structures discussed in Section 6; higher levels of magnification are required, and an extraction may be necessary. Third, residue origin must be determined to be either environmental (weathering, patination, natural degradation) or anthropogenic. Interpretation becomes increasingly important from this stage on as the analyst must infer the meaning and authenticity of the identified components present within the residue.

The second portion of the interpretive model is narrower in focus and may require combinations of high-powered microscopy, optical and SEM, as well as chemical analysis. The first of these determines if the residue is related to plant or animal sources and the second establishes whether a specific tissue can be identified. The third helps ascertain if a taxonomic designation is

possible, but the identification of taxonomy should be treated with extreme caution when employing a single methodological approach. The employment of a multi-analytical approach provides numerous lines of evidence and a stronger overall interpretation.

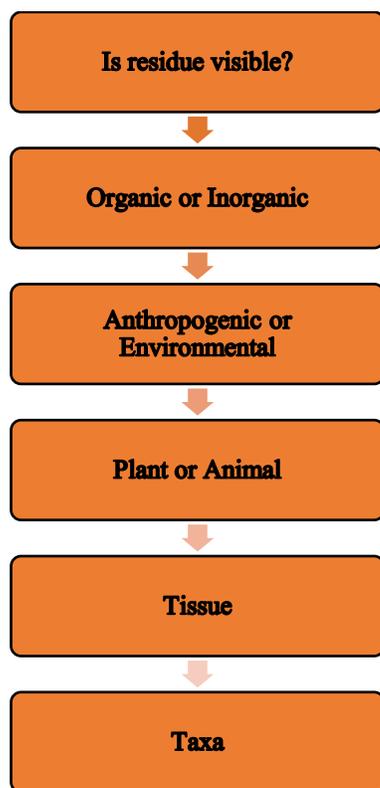


Chart 1. *Residue analysis interpretive process (Matheson and Veall 2014).*

8.0 Factors affecting the determination of function and source material

Overall, there are two main factors that affect the determination of function and source material. These include complicating factors, and limitations.

Complicating factors include the presence of multiple or overlapping wear patterns, artifact curation practices (re-use, re-sharpen, recycle), and residue mixtures. Within use-wear analysis, it is not uncommon to observe wear patterns that contain traits

common to multiple functions or materials. The differing patterns can be intermixed in varying ratios across the edge, or may be superimposed on one another. Detection of these patterns indicate multi-purpose or generalized tool use rather than discrete or specialized purposes. Pre-depositional modification or curation processes such as re-sharpening or recycling also affect the accrual of wear and may result in multiple specific wear types being present in different locations on a single artifact. Observations of this kind affect the interpretation of the use-life of the tool, as well as the use of raw lithic materials by the occupants. Lastly, residue mixtures cause complications specifically to chemical analysis. In instances of non-destructive sampling, only a miniscule amount of the sample is tested. Should the specimen consist of a mixture of residues, the possibility exists that the sampled portion may not be representative of the residue as a whole, and may provide varying results if sampled multiple times. In addition, spectroscopic chemical analyses determine matches through best-fit comparisons. The chromatogram of a mixed residue may not elicit any matches with high probability due its inclusion of multiple and probably fragmented signatures. In these instances, interpretation requires additional effort from the researcher to tease out the multiple signatures based on the ordered fragmentation patterns visible within the chromatogram. It should be noted that the state of visible compounds is greatly affected by the preservative properties of the burial context. While certain environments are capable of producing remarkable results (arid, clay-rich, etc.), others have significantly limited preservative properties that result in the degradation of organic compounds beyond the point of visible detection (i.e. podzolic environments).

Limitations include a lack of terminological standardization,

quantification and reproducibility in the field as well as the differing rates of degradation that affect chemical analysis. Although the overall uniformity of terminology has improved over the past decade, differences in the terms used for flake scar morphologies and termination still exist. Problems with quantification and reproducibility have likewise decreased significantly, and continue to do so with the increased use of stringent lab protocols and careful recordings of methodological approaches and analyses. The differing rates of degradation affect GC-MS analysis in particular (Eerkens 2005). To date, the effects of these differing rates have not been well researched in relation to microscopically visible structures.

9.0 Conclusion

The functional analysis of stone tools is integral to understanding past lifeways and developing accurate interpretations of site use, technological advancement, and human behavioral adaptations to dynamic landscapes. While the data included within this paper do not represent an exhaustive review of methodological, analytical, or interpretive approaches to the micro-analytical study of stone tools, it is hoped that this research provides a comprehensive introduction to the field. The incorporation of any of these techniques into a broader analysis provides a means to infer organic materials, a topic that is particularly important for study areas that lack organic materials. Engaging in cautious interpretation, based upon stringent methodological protocols and multiple lines of evidence aid in overcoming the limitations inherent within each approach. In addition, they also minimize biases concerning tool function based solely on morphological variability. The benefits of a coordinated, multi-analytical approach outweigh any

limitations and will ultimately provide a stronger overall interpretations. Although these approaches often focus on smaller sample sizes, they can be compared to similar studies at a macro or intra-site level. The increased use of these approaches will have implications for both regional and local resource exploitation and the adaptation of subsistence strategies through time.

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Table 2. *Various approaches taken to use-wear and residue analysis*

Method	Type	Focus	Lithic Type	Cleaning Method	Microwear Results	Residue Results	Reference
L	E	U	Flint	Not included	antler, bone, wood, fish, plant, meat, skin; post-depositional, cutting, scraping, sawing, drilling	N/A	Tringham et al 1974
L, IT	A	B	Various	Not included	Hafting, scraping, cutting; bone, hide, meat, wood	Duck, deer, rabbit, dog, rodent, cat, bear, human	Petraglia et al 1996
H	E/A	B	Obsidian	Not included	cutting, slicing; plant materials	Starch	Barton et al 1998
L/H	E	U	Flint	10% HCl acid immersion and acetone cleaning	Hafting and prehensile wear	N/A	Rots 2005
L/H	A	B	Not included	Not included	Wood, woody tissue, skin, reed, bone, soft plant, ochre	Taro, yam: starch, cellulose, ochre	Fullagar et al 2006
L/H	A	R	Not included	N/A	N/A	Ochre, resin	Lombard 2006
H	E	R	Quartz, quartzite, honrfels, dolerite, chert, chalcedony	N/A	N/A	Hair, collagen, tissue, fat, cartilage, ochre, epidermal cells, and woody tissue	Wadley and Lombard 2007
L/H, FTIR	E/A	B	Flint, Obsidian	Water and soap wash; diluted acid and base washing; demineralized water sonication	Antler; Bone; Fleshy Tissue; Hide; Tendons; Shell; Teeth	Adipocere; Calcite; Bone; Lipids; Proteins	Cesaro et al 2012
L/H	A	B	Flint	Soap and warm water wash; demineralized water sonication 5 min.	Cutting, Scraping, Whittling, Mixed; Fleshy tissue, hide, wood, soft/medium/hard materials; herbaceous plants or soft wood	N/A	Lemorini et al 2014
L/H	A	B	Flint	Liquid soap sonication; water sonication	Hideworking, soft plant cutting, butcher meat, scrape hides, process bone/antler, penetrate game	N/A	Miller 2014

L/H	E	U	Glass	Not included	Impact damage	N/A	Iovita 2014
H	E	U	Chert	Water/detergent wash, 30% NaOH sonication, rinse with water	Dry Hide	N/A	Lerner 2014
L/H	E	U	Flint, Chert, Obsidian	Not included	Trampling damage	N/A	Schoville 2014
L/H	E	U	Chert	Not included	Various, trampling, post-depositional	N/A	Wiederhold and Pevny 2014
CLSM, H	A	U	Quartz	Mild detergent sonication, 5% HCl soak	Cutting, sawing; soft to medium, medium to hard materials	N/A	Derndarsky 2006
CLSM	E	U	Flint	Water and detergent brush, 10% H5NO, water bath	Antler, wood, fresh and greasy hide, dry hide, unused	N/A	Evans and Donahue 2008
CLSM, L	E/A	B	Chert	Soap and water, swabbed with alcohol prior to imaging; one hour soak in 5% HCl.	Hard material, wood, soft plant, hide, meat, soft material, unused	N/A	Stevens et al 2010
CLSM, L/H	E	U	Chert	Water and detergent brush, 10% HCl and NaOH bath, water bath	Antler, wood, dry hide, meat, wheat; cutting and scraping	N/A	Macdonald 2014
CLSM	E/A	U	Flint	30% H2O2 soak, clean with soapy water	Sickle use; wild/semi-green and domestic/ripe plants	N/A	Ibanex et al 2014
CLSM, H	E	U	Chert, Obsidian	Detergent wash, 15% HCl bath, 15% NaOH bath	various	N/A	Stemp 2014
SEM, L/H	E	U	Quartz	sonication with 5% H2O2, detergent, water	Hide, wood, plant	N/A	Knutsson 1988
GC/MS	A	R	Ceramics	hydrolysis of powdered samples	N/A	Amino acids, proteins	Evershed and Tuross 1996
SEM, H	A	R	Ceramic	digestion in heated 50% HN03, dilution, centrifugation, and mounting	N/A	Maize and beans	Boyd et al 2006
SEM, L, EDS, GC/MS, PY	A	B	Wood and bone	Not included	Carving, diggish, awls	Beeswax, egg, vegetal fibers, castor beans	D'Errico et al 2012

H, SEM, EDS, FTIR, CRM	A	B	Chert	N/A	N/A	Bitumen, authigenic minerals	Monnier et al 2013
SEM	E	U	Flint, Obsidian, Quartzite	Not included	Cutting, skinning, scraping, sawing; flesh, bone, skin, wood, grass	N/A	Olle and Verges 2014
SEM, L/H	E	B	Chert	Detergent sonication, acetone sonication	sawing, scraping, drilling, cutting, striking; bamboo, coconut, acacia, shell, skin, meat, bone, sinew;	Yes	Borel et al 2014
SEM, L	E/A	U	Quartz	soap and water wash, water sonication	Cutting, scraping; wood, tissue, plant, medium and medium-hard materials	N/A	Lemorini et al 2014
FTIR, ATR	E	R	Various	not included	Thrusting, scraping	Muscle, bone, fat	Prinsloo et al 2014
GC/MS, L	A	R	Metal	distilled water, ethanol, acetonitrile mixture	N/A	Incense	Crowther et al 2014
BT, H, GC/MS, AS	A	R	Adzes	distilled water, ethanol, acetonitrile mixture	N/A	Resin, woody tissue	Cook 2015
SEM, H, BT, GC/MS	E/A	B	Quartz and amethyst	Water, ethanol, acetonitrile sonication	Pending	Pending	Bouchard <i>n.d.</i>

Abbreviations:

L - Low-powered Microscopy
H - High-powered Microscopy
CLSM - Confocal Laser Scanning Microscopy
SEM - Scanning Electron Microscopy
IT - Immunological Testing
FTIR - Fourier Transform Infrared Spectroscopy
ATR - Attenuated Total Reflectance
AS - Absorbance Spectroscopy
GC/MS - Gas Chromatography coupled Mass Spectroscopy
BT - Biochemical Testing
EDS - Energy Dispersive Spectroscopy
PY - Pyrolysis
CRM - Confocal Raman Spectroscopy

E - Experimental
A - Archaeological

U - Use-wear
R - Residue
B - Both

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CHAPTER THREE

EARLY PRE-CONTACT USE OF ORGANIC MATERIALS WITHIN THE NORTH SUPERIOR REGION: INDIRECT EVIDENCE THROUGH USE-WEAR ANALYSIS

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CHAPTER INTRODUCTION

This chapter serves as a case study for the use-wear portion of this thesis, one of the two primarily analytical approaches employed. The focus of this chapter is the indirect identification of organic materials at the Early Holocene site, WP11. Non-projectile artifacts including formal, informal, and expedient unifacial tools were selected for this analysis in order to better demonstrate resource use without the bias common within analyses limited to the study of projectiles.

While the discussion is the primary focus of this chapter, brief descriptions of the study area and methodological protocols are provided first. Within the discussion section, primary interpretations were divided further into manners of use (i.e. push-pull, pull, push, cut, pull-cut), and contact material hardness (CMH; soft, medium, hard). Contact material hardness was then narrowed down further through the use of higher-powered magnification. Results are further separated according to the manufacturing type of each artifact: formal, informal, or expedient. This separation allows for a clear discussion of similarities and differences between each manufacturing type. Technological variations observed are discussed, including manners of use, generalized or specialized uses, and hafting styles employed. All analyzed images and a table summarizing recorded data are included in Appendix B.

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ABSTRACT

The lack of macroscopic organic materials at Early Holocene archaeological sites in Northwestern Ontario limits conventional interpretations of subsistence strategies and resource exploitation of the early inhabitants. Use-wear analysis was used to analyze formal and expedient Early Holocene artifacts from the Upper Great Lakes region of Canada to identify and interpret the use of these otherwise invisible traces. The findings of this research indicate the specialized use of high-quality, formal artifacts; the hafting of intentionally shaped expedient artifacts used for multiple purposes; and the general, non-specified use of minimally shaped expedient artifacts. Wear patterns are indicative of dry hide, bone, meat, grassy and woody plant materials, and wood. Broader applications of the technique across the region will aid in the documentation of resource adaptation and subsistence use.

1.0 Introduction

Human use of organic materials, in the Early Holocene for subsistence and tool-manufacturing represents a major component of recorded hunter-gatherer subsistence models (Adovasio et al. 2014; Miller 2014). Material evidence of perishable technologies related to these activities is scarce within the archaeological record, in all but the most exceptionally preserved environments. The scarcity is compounded in boreal ecozones within North America due to the poor preservative properties of the soil (Hurcombe 2008; Odell 1980). This poor organic preservation has resulted in a heavy bias toward lithic artifacts in most Early Holocene assemblages, and a disproportionate amount of research directed into faunal over floral resource exploitation (Gero 1993).

Lithic microwear analysis provides a means to infer the use of Early Holocene perishable

technologies otherwise invisible in a lithic assemblage (Loebel 2013; Miller 2014; Soffer 2004). Understanding the function of lithic tools is integral to building an understanding of the lifeways of past peoples (MacDonald 2014). Microwear analysis provides this understanding through extensive analyses of both microchipping and microfeatures including polish and striations on working edges and non-working surfaces of utilized artifacts. Damages such as these have been shown to relate directly to both the manners of use and the materials that were processed (Keeley 1980; Lawn and Marshall 1979; MacDonald 2014; Odell 1979; Tringham 1974; Vaughan 1981). Contrary to the initial debate between low- and high-powered microscopic analyses, methodologies now frequently include both standards as a minimum. Modern methodological combinations include light microscopy with Fourier Transform Infrared

Spectroscopy (FTIR) (Cesaro and Lemorini 2012), confocal laser scanning microscopy (Evans and Donahue 2008; Stevens et al 2010), Scanning Electron Microscopy (SEM) (Borel et al. 2014; Bouchard 2016), and biochemical residue analyses (Ollé and Vergès 2008) among others (Van Gijn 2014).

This research is a study of lithic microwear from a collection of artifacts from an Early Holocene site in the Upper Great Lakes region of North America. The study focuses on the analysis of unifacial implements with an emphasis on organic material use, including plant and wood processing. The project utilized light microscopy with both high- (100x to 500x) and low-powered (20x to 65x) magnification.

2.0 Materials and Methods

2.1 Electric Woodpecker II

Artifacts analyzed in this study were excavated from the Electric Woodpecker II site (WPII; DdJf-12) in the Thunder Bay Region of Northwestern Ontario, Canada (Fig. 1). The current study is the first to be completed on the assemblage. The Woodpecker II site is one of five archaeological sites located approximately 30 km

east of Thunder Bay excavated by the consulting archaeological firm, Western Heritage, between 2010 and 2012 (Bennett 2015; Gilliland 2012; Gilliland and Gibson 2012; Langford 2015; McCullough 2015; Norris 2012). Though these sites currently lie inland from the northern shoreline of Lake Superior, paleogeographic reconstruction places the relict shoreline of Glacial Lake Minong at geographically contemporaneous level with this string of sites (Burwasser 1977; Julig et al. 1990; Shultis 2012; Phillips, 1982). Accelerator Mass Spectrometry (AMS) Radiocarbon dates place occupation at 9760-9540 cal yr BP (Gilliland and Gibson 2012). The extensive use of Gunflint formation as a source for lithics, parallel oblique flaking patterns, and the association of the site with middle to late stages of Lake Minong place the Woodpecker II site in both the Paleoindian Lakehead Complex and the Interlakes Composite (Bennett 2015; Bouchard 2016; Fox 1976; Fox 1980; Hinshelwood 2004; Langford 2015; Markham 2013; McCullough 2015; Ross 1997; Shultis 2013).

The tool assemblage is similar to that of other local Early Holocene sites in the Thunder Bay region, with a high occurrence of retouched

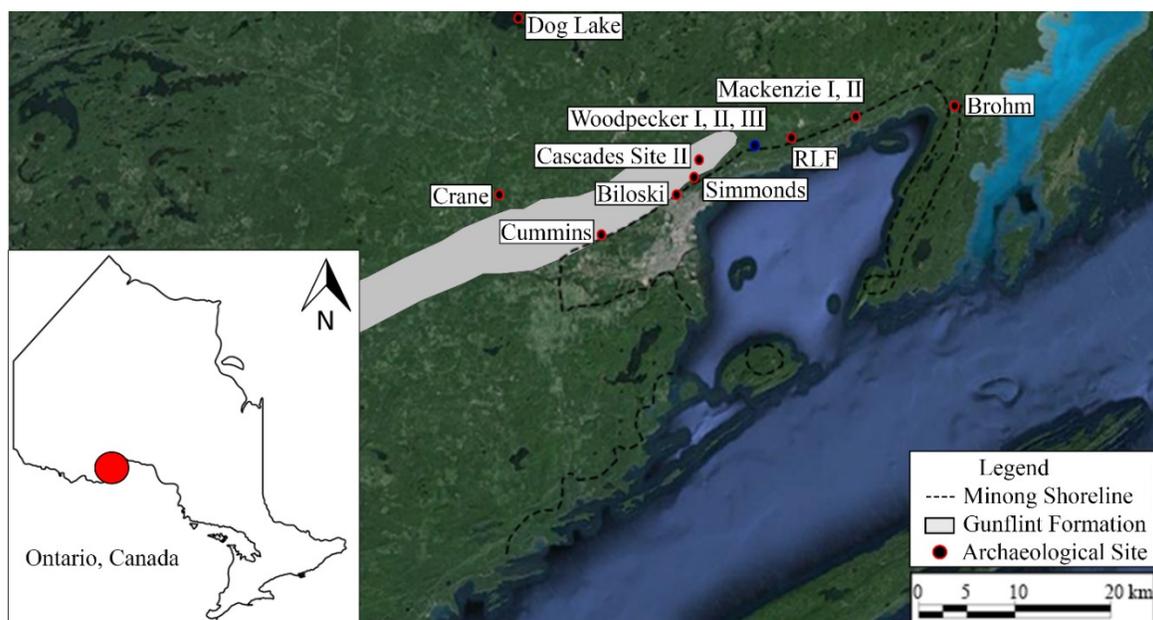


Fig. 1. Overview of the study area showing approximate locations of several Early Holocene sites within the Thunder Bay Region.



Fig. 2. The artifacts analyzed in this study to demonstrate the morphological variation amongst the unifacial artifacts.

flakes, unifacial, and bifacial artifacts, and debitage over formal tool types such as points and bifaces (Julig 1994). The inter and intra-morphological variability amongst the unifacial assemblage is high, and includes a large percentage of expedient flake tools, both with and without intentional shaping, as well as a lower percentage of formal artifact types. Tool morphology within each of these categories is not uniform (Fig. 2).

The majority of artifacts recovered from the WPII site are made of taconite, a locally available, iron-rich silicate found within the Gunflint Formation in the northern Lake Superior region. Many of the artifacts selected for this study consist of this material, with only two artifacts made of locally available chert. At the time of analysis, the full site inventory was being catalogued; artifacts selected for this study were from a subset of macroscopically identifiable specimens selected for study based on the presence of visible use-wear.

At the time of writing, very few microwear experiments using taconite have been completed. For this reason, a series of experiments were completed prior to this study to create a basic

reference collection of processual wear (Hodgson 2016a). A series of tasks were completed with 35 replica tools. These tasks included unidirectional and multidirectional scraping, planing, cutting, and carving. The experiments were performed on locally available materials including fresh and seasoned bone and wood, fresh and dry hide, soft and woody plants, and meat. Post-depositional wear experiments were completed following the preliminary analysis, and included trampling, water and sand, and water and gravel erosion (McBrearty 1998; Tringham 1974). The analysis was completed using a portable digital microscope AM4815ZTL (Dino-Lite Edge) to record images of wear accrual at several stages throughout the completion of the experimental task.

A total of 32 unifacial tools were analyzed for this study, representing approximately half of the total unifacial artifacts excavated. The collection was divided into three categories: expedient with intentional shaping; expedient with minimal shaping, and formal (Table 1). The artifacts were then analyzed to determine both manner of use and functional categorization based on wear

Table 1

Categorization of unifaces from Woodpecker II

ID #	Context- Unit	cm dbs	Artifact type	ID #	Context – Unit	cm dbs	Artifact type
UN1	489N/534E-SE	0-5	Expedient 2	UN18	509N/545E-SE	15-20	Expedient 1
UN2	490N/534E-NE	0-5	Expedient 2	UN19	512N/522E-NW	15-20	Expedient 1
UN3	495N/533E-NE	20-25	Expedient 1	UN20	513N/538E-NW	10-15	-
UN4	496N/529E-SE	5-10	Expedient 1	UN21	514N/521E-NE	15-20	Formal
UN5	500N/550E-NW	35-40	-	UN22	514N/542E-SE	35-40	-
UN6	502N/536E-SW	20-25	Expedient 1	UN23	516N/540E-NE	10-15	Formal
UN7	503N/521E-SE	10-15	-	UN24	516N/544E-NE	20-25	Expedient 1
UN8	504N/547E-NE	115-120	Expedient 1	UN25	517N/540E-NW	20-25	Expedient 1
UN9	505N/504E-SW	10-15	Expedient 1	UN26	518N/539E-SE	15-20	Expedient 1
UN10	505N/519E-SE	15-20	Formal	UN27	518N/539E-SW	5-10	Expedient 2
UN11	505N/546E-SW	100-105	Expedient 2	UN28	522N/546E-NE	30-35	-
UN12	505N/550E-SE	20-25	-	UN29	525N/543E-SW	40-45	-
UN13	507N/546E-SE	30-40	Expedient 1	UN30	526N/542E-SE	0-5	Formal
UN14	509N/518E-SE	15-20	Expedient 1	UN31	527N/540E-SE	0-5	-
UN15	509N/529E-NW	5-10	Expedient 2	UN32	557N/576E-NE	50-55	-
UN16	509N/529E-NW	15-20	-	UN33	514N/537E-NE	20-25	Formal
UN17	509N/539E-NE	25-30	-				

distribution and contact material hardness, respectively.

A collection of 32 artifacts may not be representative of the site as a whole, as the smaller size is subject to sampling bias. In order to address this issue and to document additional information on the subsistence and resource exploitation of North Superior peoples, a complementary multi-analytical residue analysis was completed (Hodgson 2016b).

2.2 Microwear Analysis

Microscopic examinations at 40x to 50x magnification using incident light microscopy were completed on all artifact edges to confirm tentative working edge locations and document any *in-situ* residues prior to cleaning. Working edges were next sonicated in an acetonitrile/ethanol/water mixture for 45 minutes and re-examined using similar magnifications. The extracted solutions were analyzed in a different study (Hodgson 2016b). Each working edge was recorded in detail prior to further examination at 100x, 200x, and 500x

magnification in discrete locations (Van Gijn 2014). Image stacking was completed with ZereneStacker© software to ensure adequate clarity of each image.

Three to six micrographic locations were selected from the dorsal and ventral surface of each artifact, including both lateral and central locations, for in-depth flake scar and feature analyses. Flake scar analysis included the characterization of flake scar attributes, including size, orientation, distribution, shape, and termination type. Kooyman (2000) recommends that scars over 4 mm in size be discounted as retouch. Scars over 2 mm in size were discounted within the present study, in accordance with observations made during prior experimental analysis (Hodgson 2016a). Subsequent feature analyses recorded the presence and degree of nibbling, crushing, rounding, smoothing, polishing, striations, and snap fractures from each selected micrograph. Higher magnifications were used to record the degree, orientation, and type of polish, striations, or hafting wears when the

Table 2

Microwear results from Woodpecker II

ID #	Artifact type	Used	Motion	Direction	Worked material	Hafted
UN1	Expedient 2	X	Push-pull	bidirectional, longitudinal	Fresh wood, fresh bone	No
UN2	Expedient 2	X	Cutting	bidirectional, transverse	Soft plant, woody plant	No
UN3	Expedient 1	X	Pull	unidirectional, longitudinal	Soft plant	Male
UN4	Expedient 1	X	Push-pull	bidirectional, longitudinal	Seasoned wood and bone	Male
UN6	Expedient 1	X	Push-pull	unidirectional, longitudinal	Dry hide	Juxtaposed
UN8	Expedient 1	X	Cutting	bidirectional, transverse	Dry wood	No
UN9	Expedient 1	X	Pull	unidirectional, longitudinal	Dry wood	Male/Juxtaposed
UN10	Formal	X	Push-pull	bidirectional, longitudinal	Fresh bone	Male
UN11	Expedient 2	X	Cutting	bidirectional, transverse	Fresh bone, meat	No
UN13	Expedient 1	X	Pull	unidirectional, longitudinal	Fresh bone, fresh wood	No
UN14	Expedient 1	X	Pull	unidirectional, longitudinal	Fresh bone, meat, dry wood, soft plant	Male
UN15	Expedient 2	X	Pull-cut	unidirectional, transverse	Fresh bone, meat	No
UN18	Expedient 1	X	Cutting	bidirectional, transverse	Fresh bone, meat	Male
UN19	Expedient 1	X	Pull	unidirectional, longitudinal	Fresh hide, soft plant	Male
UN21	Formal	X	Pull-cut	unidirectional, transverse	Dry bone, dry wood	Male
UN23	Formal	X	Pull	unidirectional, longitudinal	Fresh hide	?
UN24	Expedient 1	X	Pull	unidirectional, longitudinal	Dry wood, bone	Wrapped
UN25	Expedient 1	X	Pull-cut	unidirectional, transverse	Soft plant, meat	No
UN26	Expedient 1	X	Pull-cut	unidirectional, transverse	Dry wood, dry bone	No
UN27	Expedient 2	X	Push	unidirectional, longitudinal	Dry bone, dry wood	No
UN30	Formal	X	Push	unidirectional, longitudinal	Dry hide	Juxtaposed
UN33	Formal	-	-	-	-	Juxtaposed

presence of such was indicated via the lower magnifications.

3.0 Results

The presence of wear is influenced directly by the manner in which a tool is held and in which motion it is used (Odell 1980). Singular or multiple material tasks can be detected through the identification of overlapping wear types (Tringham 1974).

Microwear traces were successfully analyzed on 22 of the 32 artifacts included within this study (Table 2). The shorter cleaning times employed in the effort to preserve authentic, *in-situ* residues failed to provide an adequately clean working edge on nine artifacts, preventing a complete microwear analysis at this time. Post-depositional wears were identified through comparative analysis from both the literature and experimental research, and subsequently were excluded from the analysis (Hodgson 2016a; McBrearty et al 1998; Tringham 1974). Evidence indicative of hafting including micro-scarring, polish, rounding, and crushing was identified on 12

artifacts, and point to wrapped, juxtaposed, and male hafting styles (Rots 2010). In a single case, left-handedness was discernible from wear distribution and ergonomic necessity.

The manner of use for each implement is described as one of the following: push-pull, pull, pull-cut, push, and cutting (Fig. 3).

Push-pull was indicated by bifacial scarring on the working edge. In the majority of cases, the contact surface contained significantly fewer scars. When both the contact and opposing aspects contained approximately the same amount and type of damage, bidirectional movement was indicated.

Pulling motions were indicated by a significant difference in wear on the dorsal and ventral aspect of the working edge. In these cases, the contact surface would have a minimal amount of scarring, while the opposite aspect had heavy wear. None of the artifacts in this category were found to have been used bidirectionally.

Pull-cut motions were characterized by transverse movement across the contact material, evidenced by wear on both surfaces. Striations

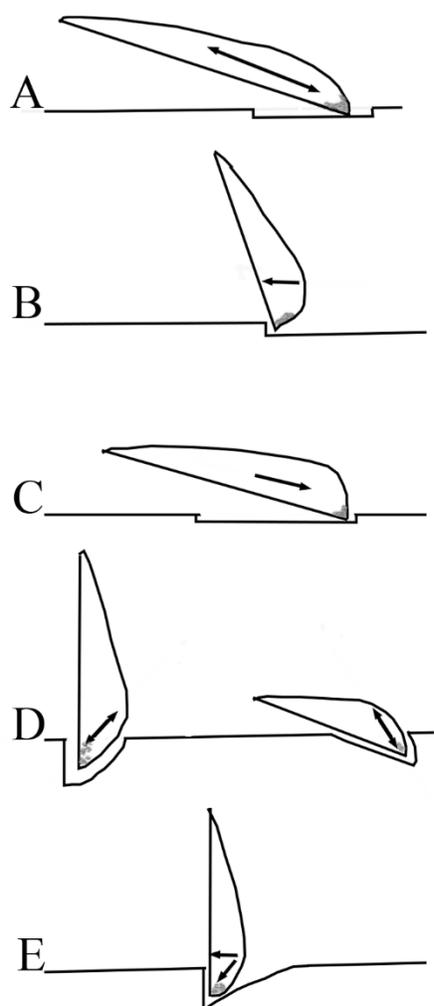


Fig. 3. Manners of use: push-pull (A), pull (B), push (C), cut (D), and pull-cut (E).

and orientation of these scars indicated unidirectional use. Pushing motions were likewise unidirectional, indicated by ventral polish, striations, nibbling, and hinged scalar scars in addition to dorsal rounding, smoothing, and stepped retouch (Fig. 4). Angle of use, acute or 90° , was indicated by wear and polish ratios on either surface.

Lastly, cutting motions were indicated by scarring on both edges with multiple orientations, multi-oriented striations, and equal amounts of wear on either surface, indicating a transverse orientation to the material.

Functional categories were determined by contact material hardness (CMH). The CMH was



Fig. 4. Wears indicative of unidirectional pushing motions including feathered scalar scars (A), ventral polish and rounding (B), and stepped retouch with incomplete step type 2 terminations (C).

characterized by the individual attributes of each of the use-related scars, in addition to the presence and degree of features including polish, nibbling, stepping, crushing, striations, and smoothing. The CMH ranged from soft to hard, with several degrees of variation in between. Comparisons with current literature and experimental data indicate both generalized and specialized use on fresh and seasoned wood, fresh or seasoned bone, dry and fresh hide, soft and

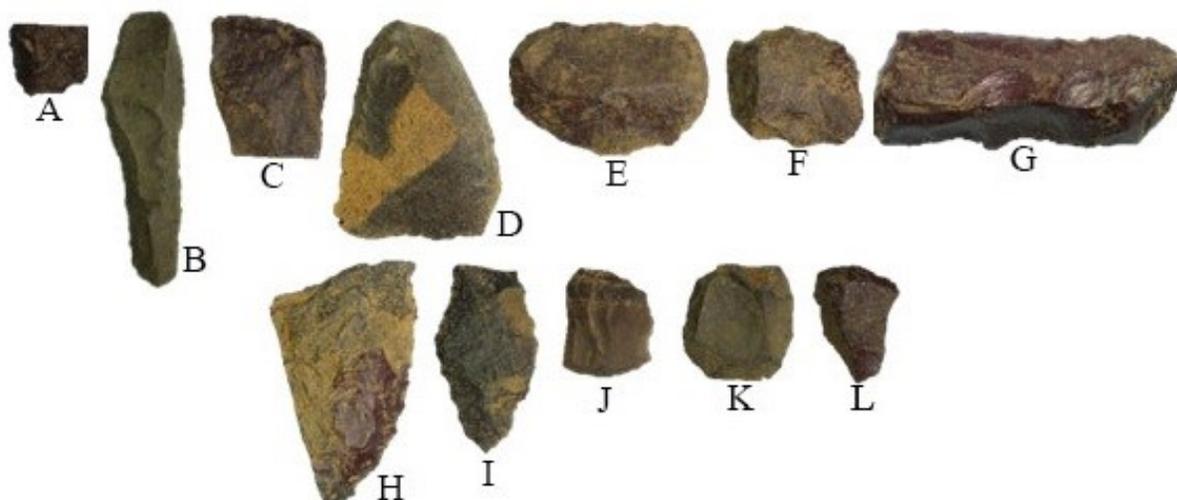


Fig 5. The 12 expediently manufactured tools with intentional shaping of which showed push-pull (A), pull (B-G), pull-cut (H, I), push (J) and cutting (K, L) manners of motion and hafting wear (A-C, E-G, J, L).

woody plant materials, and combinations of meat, bone, and hide processing. Single or multi-task and material use was indicated by overlapping wear types.

Hafting evidence included bifacial scarring or crushing of lateral edges, elongated scalar to rectangular scars with dipped or curved initiations, scar distribution along lateral tool edges, and the presence of noticeable terminations, or haft boundaries and bright spots on non-working surfaces or edges (Rots 2008, 2010).

3.1 Manner of use based on wear distribution and characterization

3.1.1 Expedient Artifacts

A total of 12 expediently manufactured tools with intentional shaping were successfully analyzed. Of these, one was used in a push-pull motion, six in a pulling motion, two in a pull-cut motion, one in a pushing motion, and two in a cutting motion. Hafting wear was present on eight, or 75%, of these artifacts (Fig. 5).

Expediently manufactured tools with minimal shaping accounted for five tools analyzed in this study. The manner of use of these artifacts was found to be fairly evenly distributed amongst the observed categories: one in push-pull, one in pull-cut, one in push, and two in

cutting. None of the analyzed artifacts in this category bore evidence of hafting (Fig. 6).

The wear accrued on expedient artifacts seems to be indicative of task-specific wear, as multiple manners of use were not identified. A division between hafted and non-hafted artifacts is apparent, with 75% of intentionally shaped expedient artifacts and zero of the non-intentionally shaped implements being hafted. This demonstrates a relationship between the task and time expenditure of utilitarian implements; expedient, lower quality artifacts intended for general and multi-task usage, and a smaller, specialized tool suite of higher quality materials meant for more specific undertakings.

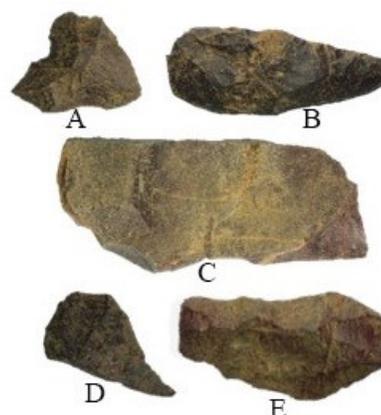


Fig. 6. Expedient artifacts demonstrating push-pull (A), pull-cut (B), push (C) and cutting (D, E) motions.

This portion of the analysis concluded that two implements were used for bidirectional scraping or planing, six for unidirectional scraping, three for unidirectional cutting, two for unidirectional planing (push), and four for transverse oriented cutting (see Table 2). A variety of hafting techniques were also employed, including juxtaposed, male, and wrapped (Fig. 7) (Rots 2005). Regardless of hafting style, the majority of hafted implements were employed in scraping or planing and required a greater input of time-cost in manufacture.

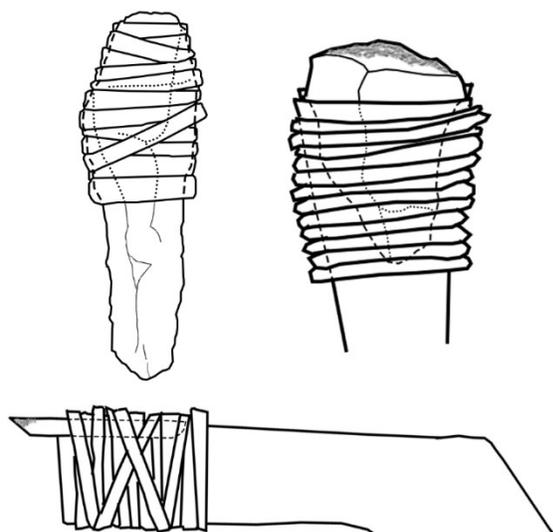


Fig.7. The hafting techniques indicated in this analysis: juxtaposed (A), male (B), and wrapped (C).

3.1.2 Formal Artifacts

Five formal artifacts were analyzed in this study. Though the number is relatively small, it reflects the overall percentage of formal to expedient tools within the total site assemblage observed throughout the sample selection process. The artifacts within this category were made of higher quality materials than those found in either expedient tool category. Mid-quality taconite represented 21 expedient artifacts, while only two were made of local and non-local high quality chert. The implements were evenly distributed among the manner of use categories, excluding UN33, and included evidence of push-pull, pull, push, and cutting. The missing working edge of UN33 prevented a positive determination of use manner.

The majority of formal uniface had evidence of hafting. Juxtaposed hafting was identified in

UN10, UN23, and UN30, with distribution indicative of an angled elbow haft present in the latter (Fig. 8). UN21 bore evidence of male style hafting (Fig. 9). Single task use was indicated for each implement within this category, primarily consisting of scraping (UN10, UN23, and UN30) and cutting (UN21). The lack of a working edge on UN33 excludes the possibility of positive determination.

3.2 Functional categories based on contact material hardness

It has been well established in the literature that the observable differences in CMH is based on scar shape and termination type (Keeley 1980; Odell 1980; Tringham 1974). With the addition of feature analysis and a functional comparative database, it becomes possible to determine increasingly detailed information concerning CMH beyond the basic soft, medium, or hard designations. These categories can be further narrowed down into variations of wood, bone, antler, hide, or plant processing. With the use of appropriate references, it becomes possible to differentiate between single-task, multi-task, single material, or overlapping material wear.

3.2.1 Multiple Use

Expedient unifaces had wear indicative of multiple material contact in every manner of use. UN1 and UN4 were used for bidirectional scraping, oriented longitudinally to the contact material. The expedient type 1 artifact (UN4), displayed a high frequency of hinged triangular and trapezoidal scars and bright, unevenly distributed polish, indicative of use on seasoned bone and wood. The expedient type 2 (UN1) artifact had similar wear, with an increased amount of triangular and scalar hinged scars, indicative of use on both fresh bone and wood.

Unidirectional pull-type implements consisted entirely of expedient tools with intentional shaping. Four of these artifacts displayed combinations of wear indicative of multi-use. Combinations included fresh bone or wood, seasoned wood or bone, fresh meat and bone, and fresh hide and plant processing. Three of these tools were used for two different processing materials, while a single artifact, UN14, was found with evidence of at least four. Pull/cut manner implements included UN15,

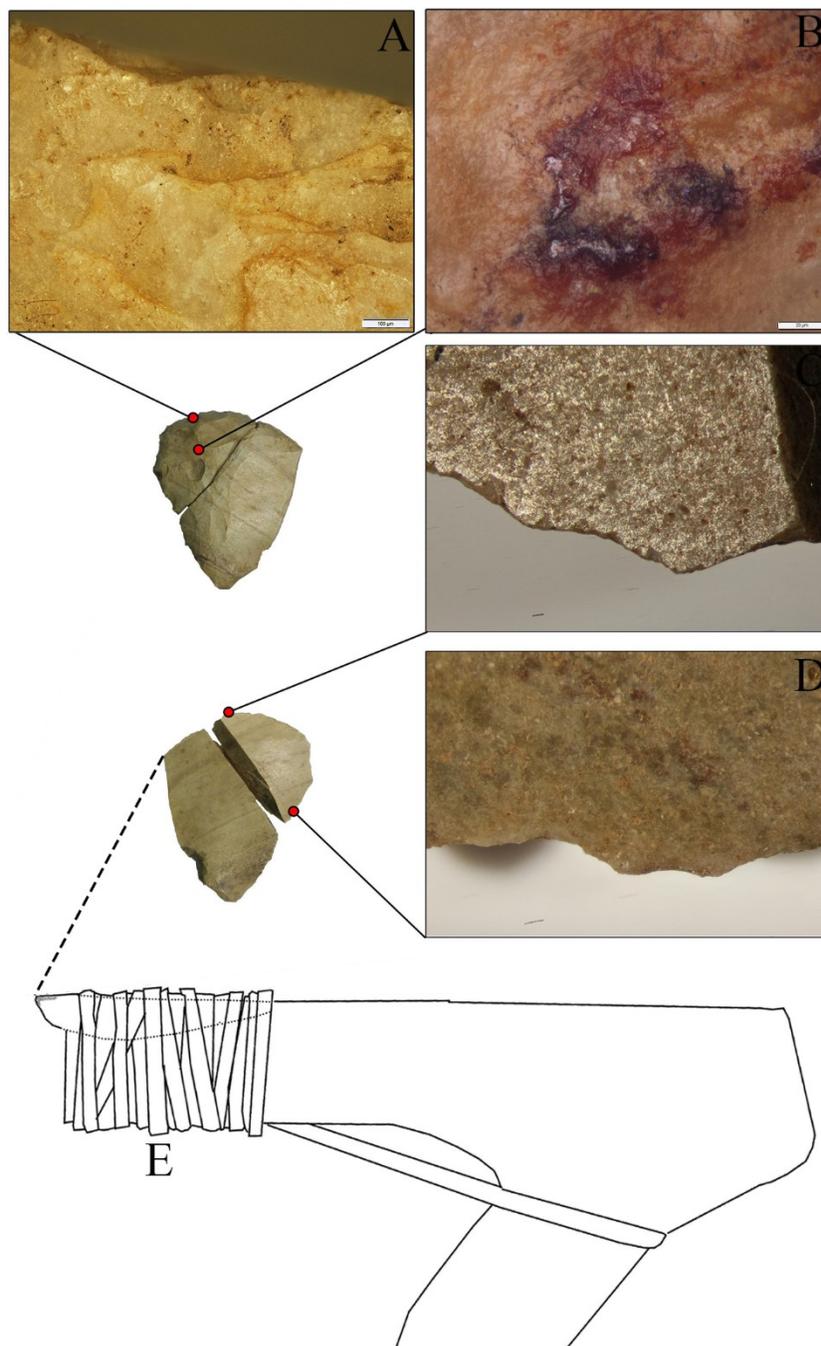


Fig. 8. Hafting and use-related wears present on UN30: heavy retouch (A), hafting residue (B), extensive greasy polish (C), and lateral nibbling and rounding (D). Possible hafting form (E) based on micro-damages.

UN25, and UN26, each of which was used unidirectionally at acute, transverse angles to the material. Each of these artifacts were employed in processing a minimum of two materials, varying between combined plant and meat

processing to seasoned wood and/or bone, as well as fresh bone and meat.

Artifact UN27 was the only expedient artifact used in a unidirectional pushing manner on multiple materials. The implement was moved longitudinally against the contact material.

Evidence is indicative of seasoned wood and bone.

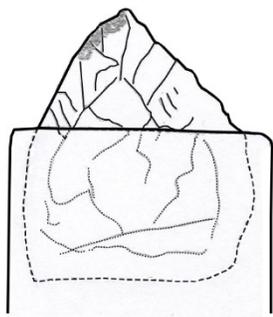


Fig. 9. An example of male hafting as seen on UN21. Use area indicated by grey.

Bidirectional cutting implements included UN18, UN2, and UN11. UN18 and UN11 demonstrate evidence of meat and fresh bone processing. Artifact UN2 displays wear indicative of plant, woody plant, and seasoned bone.

Six expedient artifacts (UN3, UN4, UN6, UN9, UN14, and UN24) bore evidence consistent with hafting on non-working edges, and evidence consistent with planing or scraping a variety of materials along the working edge. Artifact UN18, also a hafted expedient artifact, displayed wear consistent with a cutting function.

3.2.2 Specialized Use

All formal and four expedient tools displayed wear consistent with single material use. Of these nine artifacts, eight appear to have been hafted. Once again, hafting styles vary throughout.

The expedient artifacts include UN3, UN6, UN8, and UN9. Collectively, they were in contact with wood, soft plant, and dry hide processing activities. Male hafting was indicated in UN3 and UN6, while juxtaposed hafting was evident on UN9. Artifact UN8 did not display evidence of hafting.

Direct evidence of hafting, lateral crushing and/or rounding, curved scar initiations, multi-directional striations or hafting residues, was displayed on four out of five formal artifacts. The singularity, UN23, is the result of a snap fracture immediately below the working edge. Although the lower portion was not recovered, the location of the snap fracture is consistent with a snap created during the use of a hafted scraper (Shott

1995). Without the lateral edges, it is impossible to determine this with certainty, despite its likelihood. The working edge of the tool bears diagnostic fresh hide polish in the form of light rounding and weak, evenly distributed polish (Loebel 2013) (Fig. 10).

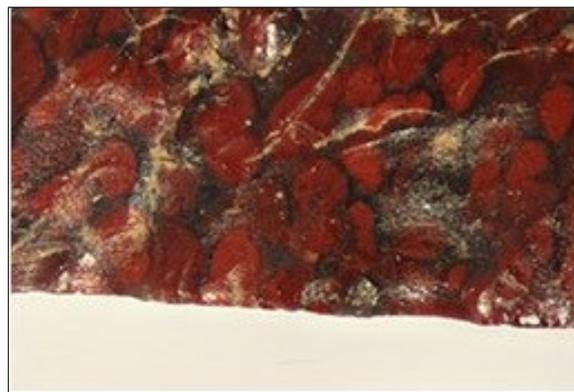


Fig 10. Feathered scalar scars, light rounding, and evenly distributed weak polish indicative of use on fresh hide.

Artifact UN10 was moved bidirectionally in a push-pull manner longitudinally across the contact material in an acute angle. Heavy, bifacial scarring with step and hinge terminations, a rough and dull polish, and clumped scar distribution indicate exclusive use on fresh bone (Maika 2012). UN21 displays similar wear patterns primarily displayed on the dorsal surface, as well as a small series of striations parallel to the working edge (Fig. 11). This indicates unidirectional cutting motions with the implement held at an acute angle to the medium-hard to hard contact material, likely seasoned bone or antler.

Artifact UN30, manufactured from chert, was employed in a unidirectional pushing manner. A heavy, greasy polish and pronounced rounding along the ventral and dorsal working edges in combination with lateral scarring, polish, and hafting residue, indicates prolonged use processing dry hide while situated in a juxtaposed, bent haft (Rots 2005). This artifact was one of the few artifacts in this study to display retouch.

The final formal artifact, UN33, was unable to be characterized down to contact material type. The analysis established that the recovered artifact did not contain a working edge due to a

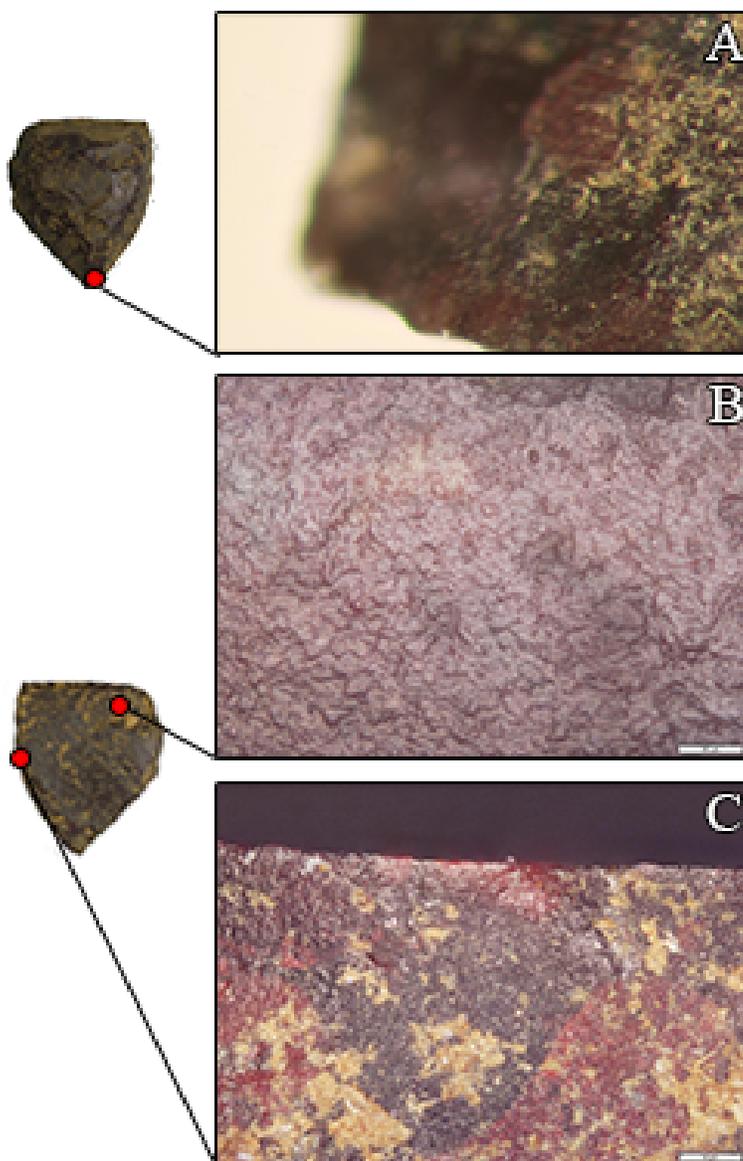


Fig. 11. Hard contact use is indicated on UN21 by striations parallel to the working edge (A), extensive dull and rough polish on the ventral surface (B), and multi-directional striations and minor lateral crushing and rounding within the hafted area (C).

previous snap fracture. The remaining portion displays diagnostic hafting evidence: bifacial scarring and very minor crushing of the lateral edges, both with similar placements; the presence of elongated scalar scars with curved initiations; and very bright spots caused by the detached microflakes rubbing against the tool edge (Rots 2006, 2010). Without the working edge it is impossible to determine what the contact material

may have been, although it is possible to deduce the artifact's complete morphology from the present evidence (Fig. 12).

4.0 Discussion

It is necessary to demonstrate actual use retouch and polish in order to correctly identify tool function (Fox 1979). Morphological and technological analyses alone may not provide

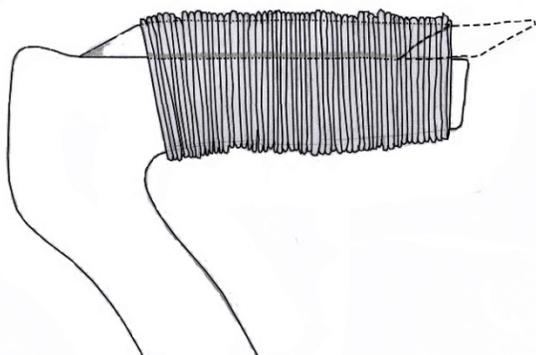


Fig. 12. Proposed hafting style of incomplete artifact UN33.

adequate data to interpret function and task of artifacts accurately. In the current study, morphological analysis alone would identify seven artifacts as traditional endscrapers, typically associated with hide working (Hayden 1979). Without additional analysis this interpretation would be inaccurate as two of these scrapers were used for cutting as opposed to scraping activities, and three additional endscrapers were used in processing wood or bone rather than hide working. The remaining artifacts, without a detailed edge analysis, could be described as retouched flakes, preforms, shatter, or cores based on appearance, despite their use for other activities. In essence, microwear analysis has helped to change the view of lithic typology, particularly in relation to functional categories (Yerkes and Kardulias 1993). The use of microwear analysis adds another layer to archaeological interpretation, allowing insight into a varied and complex tool technology. This line of evidence will allow researchers to see beyond the formal and expedient tool categories, to a frequently overlooked category of high functioning yet expedient utilitarian implements.

The availability of local toolstone has shown both an increase in expedient artifact manufacture and a decrease in retouch frequency (Julig et al 1987a). Excluding UN30, the artifacts in this study did not exhibit evidence of extensive retouch despite the significant amount of hafting, a practice that has been documented previously in the Lower Great Lake region of Canada (Erin 2012; Miller 2014).

The probability of hafting within the sample can be divided into three simplified categories: low (expedient, minimal shaping), medium (expedient with intentional shaping), and high (formal). The stark contrast between these divisions emphasizes the differential time investment for generalized and specialized tools.

Despite their lower quality of manufacture, the generalized implements were hafted approximately 75% of the time. Greater reliance on expedient technologies has been argued to be due to readily available lithic materials (Bamforth 1986), or increased sedentism (Kelly and Todd 1988), both of which may be expressed at the WPII site via readily available lithic materials and the possibility of seasonally caribou hunting at nearby crossings (Carr 2012; Fox 1975; Langford 2015; Norris 2012). Though the artifact number was small, the use of high quality lithics for single-material tasks and lower quality taconite for multi-material tasks was evident.

5.0 Conclusion

Based on morphology alone, the artifacts within this study would have provided an inaccurate account of tool use and resource exploitation at the WPII site. With the inclusion of microwear analysis in the interpretation of these artifacts, evidence of organic materials beyond hide scraping become visible. Butchering, bone processing, wood planing, whittling, or carving, and both soft and woody plant processing all took place at the WPII site. Evidence of these activities tells an increasingly rich and detailed narrative concerning the people of this area, a narrative to which all the organic material evidence is lost. Both resource exploitation and subsistence strategies can be interpreted further, in addition to perishable technologies no longer visible within the archaeological record. Additional research through alternate methodologies, such as spatial, manufacture, or residue analyses, will enable an even more complete record of otherwise invisible tool function, perishable technologies, and resource use in the Early Holocene.

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CHAPTER FOUR

MULTI-ANALYTICAL RESIDUE ANALYSES ON EARLY HOLOCENE LITHIC ASSEMBLAGES WITHIN THE BOREAL FOREST OF CANADA: A FEASIBILITY TEST AND AN EVALUATION OF RESIDUE INTERPRETATIONS

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CHAPTER INTRODUCTION

This chapter serves as a case study for the multi-analytical residue approach, the second broad analytical approach employed within this thesis. The focus of this portion of the project is threefold: to successfully obtain interpretable results from multiple lines of evidence, to determine the relative strengths and interpretive values of these methods based upon a custom-created point scale, and to determine if significant relationships exist within each specific combinations of methods, i.e. if positive results in one method are a likely indicator of positive results in others.

The results are presented following a brief description of sample size, site context and a detailed description of the methodological protocols employed within the case study. These are divided further into individual analysis sections, in which the results from each line of evidence are presented. Images of all *in situ* residues included in the interpretations are included within the chapter. Images of all extracted residues and GC-MS chromatograms can be found in Appendices C and D. A discussion of interpretative values follows. Interpretive values were based on the number of results achieved through the use of all five methodologies. Each result counted as a positive score, regardless of the reproducibility of that result

in alternate methods. The value was then marked as low, medium, high, or very high based on these scores. The reproducibility of these results is taken into account through a determination of interpretative strength. Negative, weak, positive, and strong positive strengths were noted. In order to determine if statistically significant relationships could be found between methods with positive interpretive strength, a Kruskal-Wallis H-test was used. The results of this test allowed for a recommendation of methods with higher data yields for scenarios in which time and cost factors affect the number of methodological techniques employed within multi-analytical residue analyses.

Submission Title Page: Multi-analytical residue analyses on Early Holocene lithic assemblages within the boreal forest of Canada: A feasibility test and an evaluation of residue interpretations.

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Multi-analytical residue analyses on Early Holocene lithic assemblages within the boreal forest of Canada: A feasibility test and an evaluation of residue interpretations.

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ABSTRACT

A multi-analytical residue analysis was completed on unifacially flaked lithic artifacts from the boreal forest in the Thunder Bay District of Northwestern Ontario, Canada. A detailed, multi-analytical approach provides insight into utilitarian tool use and the feasibility of the selected multi-analytical approach. The combined methods approach using microscopy, biochemical, and analytical chemical techniques was successful in identifying source materials. Findings indicate the use of both singular and multi-purpose tools on varying plant and animal sources as well as evidence of hafting materials.

1.0 Introduction

Despite the frequent occurrence of perishable materials within archaeological assemblages throughout the world, a lack of preservative properties within podzolic environments continues to prevent the discovery of these items within certain locales. The noticeable lack of organic artifacts beyond a certain age is due primarily to their vulnerability to both chemical and biological degradation (Hurcombe 2006), a condition that is particularly pronounced in the podzolic soils of Northwestern Ontario (Price and Burton 2010; Stewart 2002). However, trace evidence of organic material has been identified on inorganic artifacts from the region, primarily through microscopic and chemical analyses (Bouchard 2016; Boyd and Surette 2010; Burchill 2015; Cook 2015; Newman and Julig 1989). The present study employs several methodological approaches sequentially to determine their feasibility in lithic residue analysis.

The sample consists of a selection of unifacially flaked artifacts from an Early Holocene archaeological site near Thunder Bay, Ontario, Canada. The successful *in situ* analysis, removal and subsequent interpretation of organic material may provide insight into a range of utilitarian activities that are otherwise ‘invisible’ within the archaeological record. By focusing on this highly variable unifacial tool type rather than more frequently recognized point type tools, it is hoped that activities including hide, bone, and woodworking can be demonstrated.

Past residue analyses in the region have revealed food technologies on ceramic vessels (Boyd et al. 2008; Boyd et al. 2014; Burchill 2015), food residues on lithic tools (Speers et al. 2015), and tentative blood residues (Newman and Julig 1989). Additionally, residue analysis of lithic tools has been further developed in recent years, and has successfully indicated hafting resin (Lombard 2008; Lombard and Wadley 2006). Current research has attempted to identify

resinous materials discovered on quartz and amethyst artifacts (Bouchard 2016). Examination of trace residues such as these can provide insight into the ways in which both formal and informal artifact types of various materials were employed. Additionally, it can provide information concerning transfer residues through wear and tear, manufacture, or storage.

This study addresses the feasibility of several residue methodologies used sequentially on artifacts recovered from an Early Holocene depositional environment noted for poor organic preservation. It focuses on the use of a tri-mixture solvent solution removal approach, low and high powered microscopic analysis, colorimetric biochemical testing, absorbance spectroscopy (AS) and gas chromatography coupled mass spectroscopy (GC-MS). *In situ* microscopic analysis, a non-destructive and stand-alone methodology employed by numerous residue analysts around the world, was included as well. A summary of methodological procedures and the results from each approach are presented, as is consideration of the quality of data attainable when multiple lines of evidence are pursued. The resulting interpretive strengths and relative values are determined, and statistically significant relationships between methods are identified. Finally, recommendations are offered for the selection of methodologies in the context of time limitations.

2.0 Materials and Methods

2.1 Artifact Sample Context

The consulting archaeological firm Western Heritage provided thirty-two artifacts for analysis (Fig. 1). Of this total, 22 were successfully analyzed, and include five formal, 12 shaped expedient, and five minimally shaped expedient tools (Table 1).

The sample was excavated from the Woodpecker II site (DdJf-12) in the Thunder Bay region of Northwestern Ontario, Canada. The Woodpecker II site is one of five archaeological sites located approximately 30 km east of Thunder Bay excavated by Western Heritage between 2010 and 2012 (Fig. 2). Though these sites currently lie inland from the northern shoreline of Lake Superior, paleogeographic reconstruction places the relict shoreline of Glacial Lake Minong at geographically contemporaneous level with this string of sites (Burwasser 1977; Julig et al. 1990; Phillips 1993; Shultz 2013). Accelerator Mass Spectrometry (AMS) Radiocarbon dates place occupation within the Early Holocene, 9760-9540 cal yr BP (Gilliland and Gibson 2012).

The majority of artifacts recovered from the Woodpecker II site are made of taconite, a locally available, iron-rich silicate found within the Gunflint Formation in the Superior region. All



Fig. 1. Examples of morphological variability within the sample from Woodpecker II unifacial tool types.

Table 1
Unifacial Sample Summary

ID #	Location	Depth*	Working Edge	ID #	Location	Depth*	Working Edge
UN1	489N/534E-SE	0-5	left lateral concavity	UN18	509N/545E-SE	15-20	right lateral edge
UN2	490N/534E-NE	0-5	proximal edge	UN19	512N/522E-NW	15-20	proximal edge
UN3	495N/533E-NE	20-25	left lateral edge	UN20	513N/538E-NW	10-15	
UN4	496N/529E-SE	5-10	distal edge	UN21	514N/521E-NE	15-20	distal edge
UN5	500N/550E-NW	35-40		UN22	514N/542E-SE	35-40	
UN6	502N/536E-SW	20-25	distal edge	UN23	516N/540E-NE	10-15	n/a
UN7	503N/521E-SE	10-15		UN24	516N/544E-NE	20-25	lateral edges
UN8	504N/547E-NE	115-120	right lateral edge	UN25	517N/540E-NW	20-25	n/a
UN9	505N/504E-SW	10-15	proximal edge	UN26	518N/539E-SE	15-20	right lateral/distal edge
UN10	505N/519E-SE	15-20	proximal edge	UN27	518N/539E-SW	5-10	distal edge
UN11	505N/546E-SW	100-105	right lateral edge	UN28	522N/546E-NE	30-35	
UN12	505N/550E-SE	20-25	left lateral edge	UN29	525N/543E-SW	40-45	right lateral edge
UN13	507N/546E-SE	30-40	distal edge	UN30	526N/542E-SE	0-5	proximal edge
UN14	509N/518E-SE	15-20		UN31	527N/540E-SE	0-5	
UN15	509N/529E-NW	5-10	right lateral edge	UN32	557N/576E-NE	50-55	
UN16	509N/529E-NW	15-20		UN33	514N/537E-NE	20-25	n/a
UN17	509N/539E-NE	25-30					

*Depth is recorded as centimeters depth below surface.

artifacts selected for this study, excluding a single chert uniface, consist of this highly variable material. The results from this study have been incorporated into a broader study of Early Holocene unifacial functionality elsewhere (Hodgson 2016b).

2.2 Multi-analytical Residue Analysis

This study encompassed five methodological approaches: incident and transmitted light microscopy (100-500x magnification), colorimetric biochemical testing, absorbance spectroscopy (AS), and gas chromatography coupled mass spectroscopy (GC-MS).

Prior to residue extraction, each artifact was examined under low-powered incident light to identify potential working edges and to characterize any residue that may be present. Two removal processes were included in this analysis, removal by sonicating the selected area of the tool

and targeted spot removal. While the majority of extracted residues were removed from working edges, at all times possible hafting areas were included as well. Spot removals were attempted on artifact surfaces as deemed necessary throughout the analysis. Results from the preliminary observations can be found in Hodgson (2016a).

2.2.1 Residue Extraction

The removal solution used in this study consisted of a 1:1:1 tri-mixture of double distilled water (ddH₂O), ethanol (EtOH), and acetonitrile (ACN). The working edge and/or hafting area was submerged into a sterile glass vessel with an adequate amount of tri-mixture to cover the targeted area, leaving the remainder of the artifact untouched (Fig. 3). The vessels were then sonicated for 45 minutes. Due to the feasibility-testing nature of this study, shorter sonication

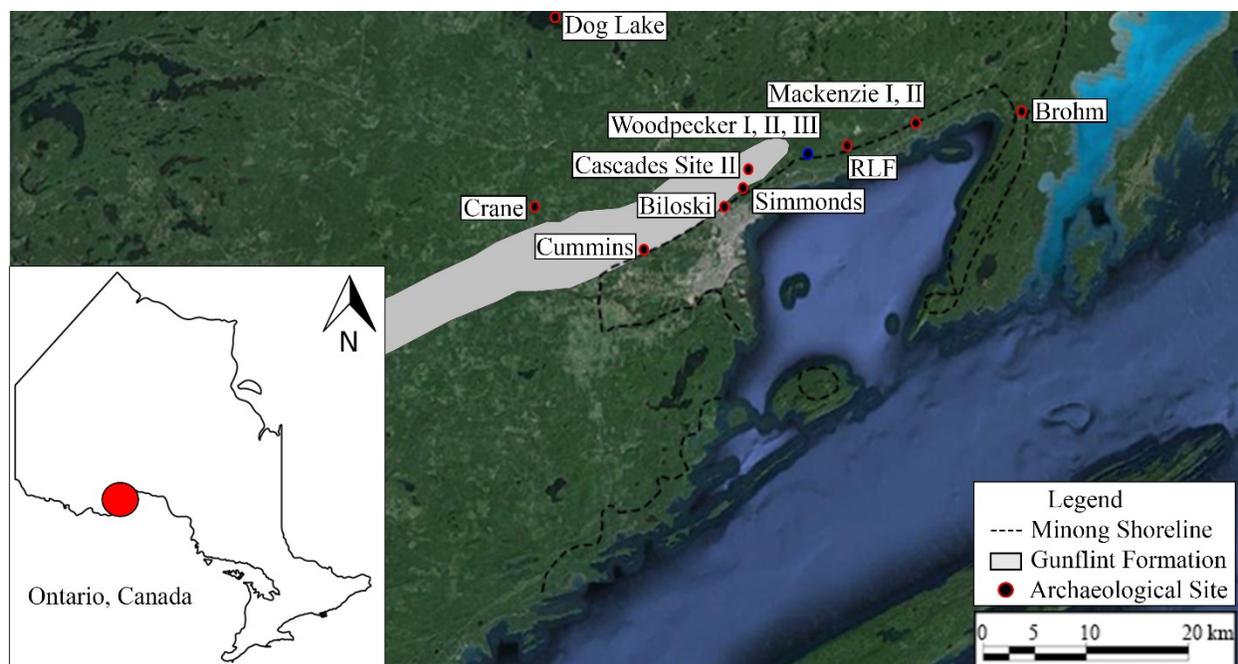


Fig. 2. Early Holocene archaeological sites along the relict Glacial Lake Minong shoreline. The study area is marked in blue.

times were chosen rather than those common in current literature to avoid the complete removal of residues adhering to the tool edge.

A tri-mixture solvent solution was selected as the primary solvent used in order to maximize extraction effectiveness. Acetonitrile and ethanol are effective at removing hydrophobic compounds such as lipids, even when mixed with water (Lin et al. 2007). The concurrent use of water in a solvent mixture increases the overall polarity, allowing for the removal of compounds damaged by oxidation over time. Acetonitrile was selected due to both its miscibility with water and its capacity to dissolve amino and fatty acids. Ethanol is likewise miscible with water, and is effective at dissolving resin acids. This tri-mixture, capable of breaking down a variety of organic residues, was ideal for non-specific feasibility determination. At this time, the practice has not become widespread within organic residue analysis (Crowther et al. 2015; Fullagar et al. 2015), but has become increasingly common amongst biomolecular studies of similar sources within biological fields (Coen et al. 2003; Kim et al. 2004; Lin et al. 2007).

Following the extraction, solutions were evaporated at room temperature to a quantity of no more than 2 ml and transferred into an acid

washed sterile 2 ml glass crimp-top vials. Portions of 0.05 ml and 0.02 ml were set aside for biochemical testing and transmitted light microscopic analysis. The 0.02 ml portion was desiccated on a sterile glass slide and mounted under a glass coverslip for later analysis.



Fig. 3. Example of residue extraction method.

2.2.2 Microscopy

In the field of archaeological residue analysis transmitted and incident light microscopic analysis began in earnest with the publication of Briuer's *New clues to stone tool function: plant and animal residues* (Briuer 1976). This was followed by rapid advances in interest concerning blood (Loy 1983; Newman and Julig 1989), starch grains (Loy et al. 1992; Shafer and

Holloway 1979), and microfossil analysis (Coughin and Claasen 1982; Piperno 1990) throughout the 1980's. Today, the field has become increasingly diversified and includes many approaches and geographical foci, and extends to a time-depth of over 1,000,000 years (Hardy and Rogers 2001). Recent studies have successfully identified a range of organic residue components including hafting resins (Lombard 2006a; Lombard and Wadley 2006; Mazza et al. 2006), starch grains (Boyd et al. 2014; Fullagar et al. 2006; Zarillo and Kooyman 2006); plant and woody tissues (Fullagar and Jones 2004; Wadley et al. 2004), phytoliths (Piperno 2006), and raphides (Crowther 2006), amongst others. Magnifications vary from low (10x – 100x) to high (100x – 1000x).

Samples were mounted to allow for adequate manipulation of the artifact, allowing 90° views of the working edges. Working edges were first examined under 100x-200x magnification, with higher magnification employed as needed. Secondary scans of possible hafting edges and remaining tool surfaces were undertaken following the initial examination. Micrographs were taken of any *in situ* residue found with an Olympus BX51 stereoscopic microscope. The location of each residue was recorded and compared with images from the existing literature as well as prepared experimental samples for identification. Prior to analysis, all micrographs were stacked using ZereneStacker© software to ensure an adequate depth of field.

Prepared slides were observed at 100x, 200x, and 500x magnification with an Olympus BX51 stereoscopic microscope. Images were recorded for all potentially identifiable particulates, including fibers, phytoliths, starches, lithic flakes, and any other possible evidence of faunal or floral contact. Synthetic fibers, and extremely well-preserved starches and pollens were noted as possible contaminants and excluded from further study.

2.2.3 Biochemical Testing

Colorimetric biochemical testing is currently used in numerous fields of study, including forensics, biochemistry, and biomedicine (Cook 2015; Fullagar et al. 2015; Matheson and Veall 2014). The tests provide direct evidence of the presence or absence of specific compounds

within organic residues through a pre-determined color change. Tests for carbohydrates, starches, fatty acids, and proteins were included within the present study, and were optimized for immediate analysis with a spectrophotometer. Sample blanks were tested and recorded for each biochemical procedure to serve as negative controls.

Limitations of this method are twofold. First, the minimum concentration threshold to indicate the presence of a compound using these tests is unknown. This is particularly relevant with the sample discussed here, as it was anticipated that the podzolic depositional environment will result in low quantities of organic substances on the tools. Second, the test cannot determine the source of positive results; if an artifact has been contaminated with starchy food particles or the blood of a careless archaeologist for example, the results of a colorimetric test will be positive. The immediate characterization of the test solution with absorbance spectroscopic techniques aids in the alleviation of these limitations.

Tests used in the study included the carbohydrate test (Kanzaki and Berger 1959; Masuko et al. 2005; Mecozzi 2005), the IKI test for starches (Briuer 1976; McCready and Hassid 1943), fatty acid test (Falholt and Lund 1973; Soloni and Sardina 1973), and the Bradford test for proteins (Bradford 1976). Observed color changes were noted and photographed when present.

2.2.4 Absorbance Spectroscopy

Absorbance spectroscopic readings were taken immediately following the biochemical tests (Matheson and Veall 2014). Two small portions (0.02 ml) of each sample solution were placed into the specimen tray of a Bio-Tech Epoch Micro Plate Spectrophotometer instrument, with up to six samples tested per run. The sample tray was cleaned with a 70% ethanol solution between tests. Baselines were determined by testing a blank sample of each test type. Positive readings were determined via frequency comparisons with blank readings of each test type.

2.2.5 Gas Chromatography coupled Mass Spectroscopy

Following initial removal, each 2 ml vial was covered in parafilm and placed into a -84° freezer for several hours until solid. Once solid, the parafilm covering were removed and the samples were freeze dried under a vacuum for a minimum of 24 hours, or until dry, in order to limit contaminants. The samples were then derivatized with 0.6 ml LCMS-grade acetonitrile (Sigma-Aldrich) and 0.1 ml of BSTFA (bis(trimethylsilyl)trifluoroacetamide) (Sigma-Aldrich). Derivatization was completed to reduce the polarity of functional groups containing oxygen and nitrogen and to aid in the separation of molecules within the column (Halket et al. 2004). The vials were purged with nitrogen, sealed by a crimped cap, and incubated at 120° C on a Baxter Scientific Multi-Block for 30 minutes. The samples were then immediately analyzed.

A Varian model 450 gas chromatograph was coupled with a Varian model 300-MS quadrupole mass spectrometer equipped with a Factor Four™ capillary column. Helium was used as the carrier gas and samples were introduced via splitless mode in an autosampler with the injection port at a temperature of 270° C. The column temperature was initially held at 50° C for 2 minutes before being increased to 155° C at a rate of 8° C per minute. Temperature was again increased to 275° C at a rate of 40° C per minute and held for nine minutes. The ion source was set at 200° C under electron ionization (EI) conditions, producing ionization energy of 70 eV. A scan range of 40 to 500 m/z was used, with a GC-MS interface temperature set at 266° C.

Output files were analyzed using Varian MS Workstation (Version 6) and the NIST98 Mass Spectral Database. Any peaks above background static were recorded. A minimal threshold was not in place due to the highly degraded environment of the study area, a podzolic boreal forest, in order to ensure the collection of even the smallest amount of data. Compounds of potential archaeological relevance were first matched to compounds from the database, when possible. When a suitable match could not be determined in this manner, the compounds were examined

manually to ensure a positive identification. Chemicals that were determined to be contaminants were noted and excluded from the resulting archaeological interpretation.

3.0 Results

The results of this study have been analyzed in the following ways: individual analysis, interpretive value, and interpretive strength. First, the individual analysis (Section 3.1) does not factor in results from each method, but rather, addresses each individually. Second, the total success of each method was given a numerical score, determining the interpretative value of each resultant data set. A ranking system such as this is imperative due to the nature of sampling; it cannot be ruled out that five methodologies all sampled precisely the same portion of a residue. Because of the nature of archaeological residues, it is strongly possible that each residue is a mixture, and that different methodological samples may represent different components of that mixture. Third, the overall interpretative strength of each sample was determined using a scale from 0 to 4, where one point is received for each consistent result.² High inter-methodological consistencies result in higher interpretative strength scores. The strength of each methodological approach was then tested as a dependent variable against every other methodology in addition to the pre-determined overall strength score. A Kruskal-Wallis H-Test was used to determine the presence or absence of statistically significant relationships amongst the data.

3.1 Individual Analyses

3.1.2 Incident Light Microscopy

In situ microscopic analyses were completed prior to and following the residue extraction. Residues directly on the cleaned artifact or appearing to lie under remaining adhering sediments were recorded as potentially authentic. These residues consisted of white, amorphous residues, translucent red residues, woody cells or longitudinally striated muscle residues, embedded fibers, and possible pitch or resin

² Note: Absorbance spectroscopy and biochemical strengths are counted together as one point due to their 100% inter-methodological consistency.

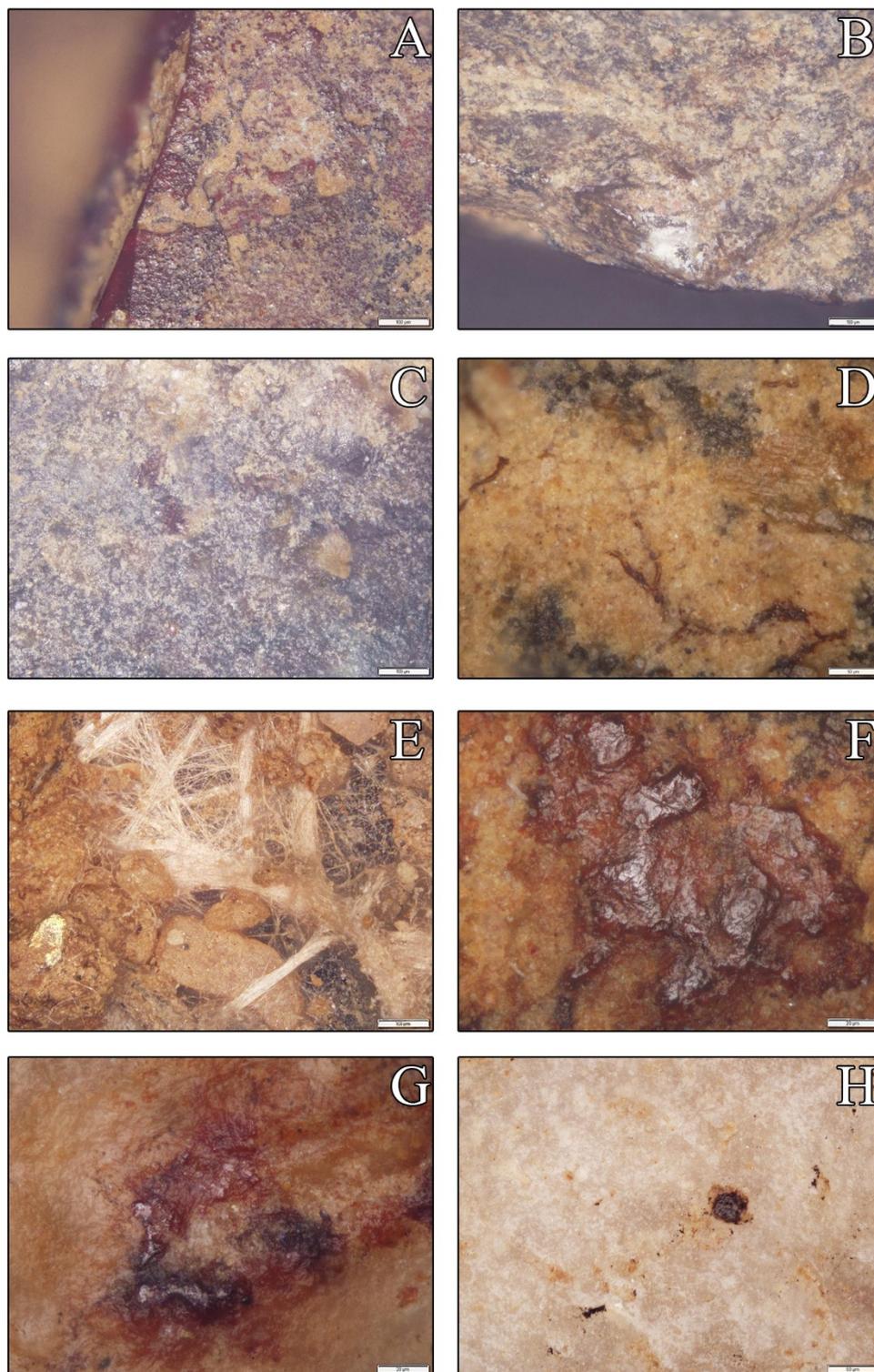


Fig. 4. Examples of residues located *in situ*: amorphous white residues (A,B); translucent red residue (C); embedded hyphae fibers (D); cluster of white fibers (E); thick, red residues with visible striations (F,G); and red/black opaque residue (H).

residues (Fig. 4). Tentative identifications of these residues were obtained by a comparison of visual characteristics with experimental and archaeological residues that exist within the current literature.

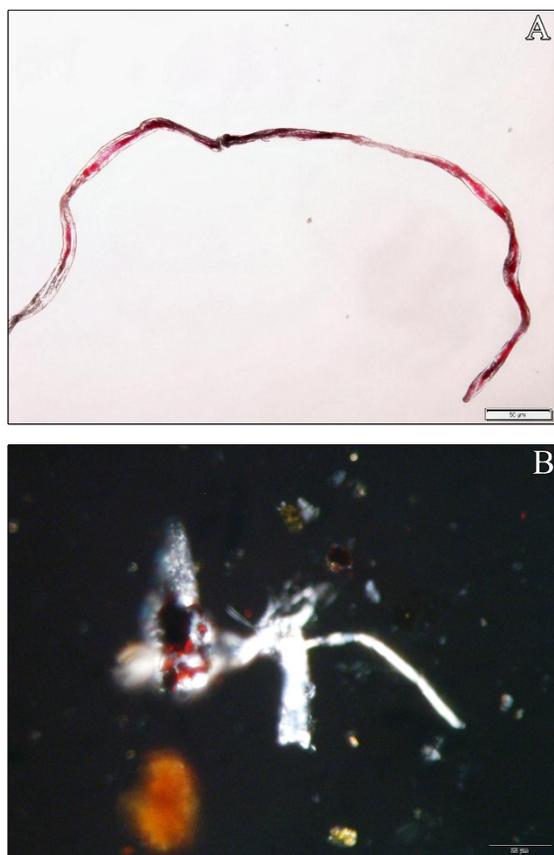


Fig. 5. Cellulosic fibers: a contaminant, degraded, dyed red fiber (A); and a degraded cellulosic fiber found in association with a taconite microflake (B).

3.1.3 Transmitted Light Microscopy

A variety of particulate materials were observed during this portion of analysis. Synthetic and dyed fibers, pollen, and starch grains, when found in exceedingly good condition, were considered to be possible contamination and excluded from further analysis. A clean slide was placed in the lab for a two-day period, mounted, and analyzed for comparison as a blank to rule out lab contaminants. Soil contaminants were ruled out through the analysis of two soil samples recovered from the burial environment.

Identified particulate materials include degraded cellulosic and collagen fibers (Figs. 5,

6), both vascular and structural plant and woody tissues (Figs. 7, 8), hematite, charcoal and burnt carbon matter (Fig. 9), a small selection of raphides and phytoliths (Fig. 10), feather and shell (Fig. 11), and a variety of microscopic lithic flakes (Fig. 12). Sample-specific results are presented in Table 2. Tentative identifications were made using a combination of comparative samples within the literature, in addition to a small selection of experimentally replicated comparisons.

3.1.4 Biochemical Tests and Absorbance Spectroscopy

It was determined throughout the testing that the acetonitrile within the tri-mixture solvent reacted positively with the Bradford protein test, and resulted in the exclusion of this test from further analysis. Faint colorimetric changes were observed in each of the remaining tests, a final summary of which can be seen in Table 3. The fatty acid test revealed a noticeable color change on eight artifacts. The starch test only tested positive on two artifacts, albeit with an exceedingly strong colorimetric change. Due to the unusually strong reaction, post-excavation starch contamination could not be ruled out. The carbohydrate test elicited positive results on six artifacts.

3.1.5 Gas Chromatography coupled Mass Spectroscopy

The strength of results within this section were determined by the number and type of compounds listed within each chromatographic spectra, and were divided into weak, positive, and strong categories (see Table 4, located at end of paper). Weak positives were indicated with one compound, positives with two to three compounds indicative of the same source, and strong positives in the case of four or more compounds indicative of the same source. The presence of diagnostic compounds, such as the terpenoid breakdown product oleanolic acid in UN18, likewise resulted in a strong positive designation.

Strong results were interpreted from nine of the 23 extractions. Of these, five indicated positive contact with plant material, two with a combination of plant and burnt organic material, one with animal, and one with a combination of

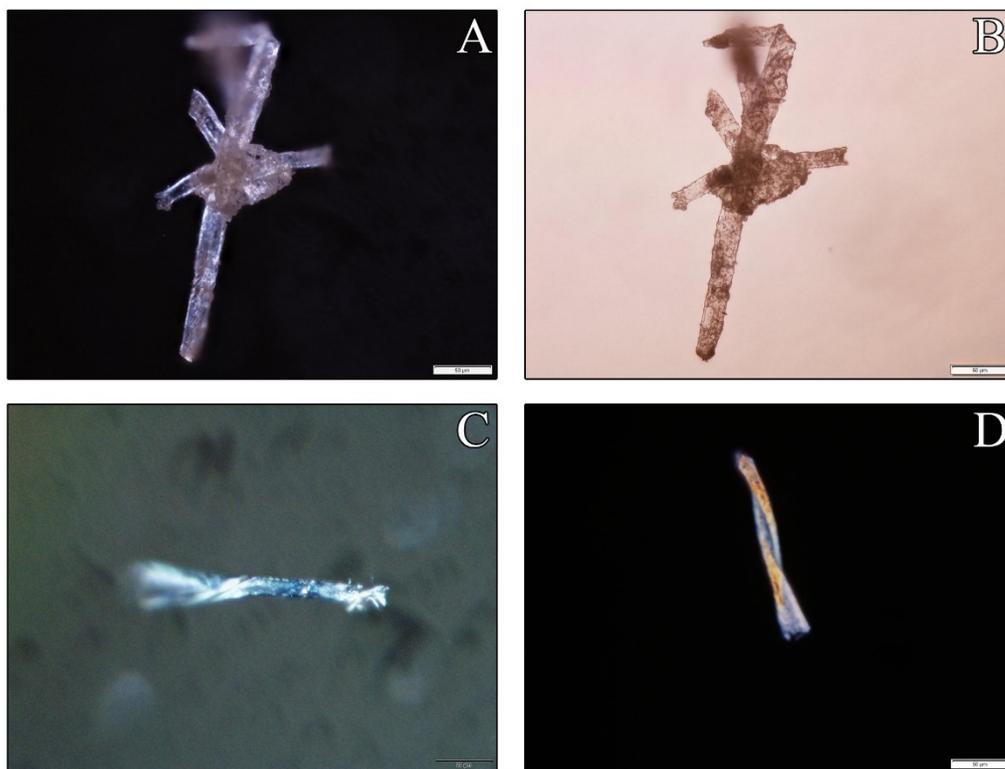


Fig. 6. Collagen fibers identified within the sample study.

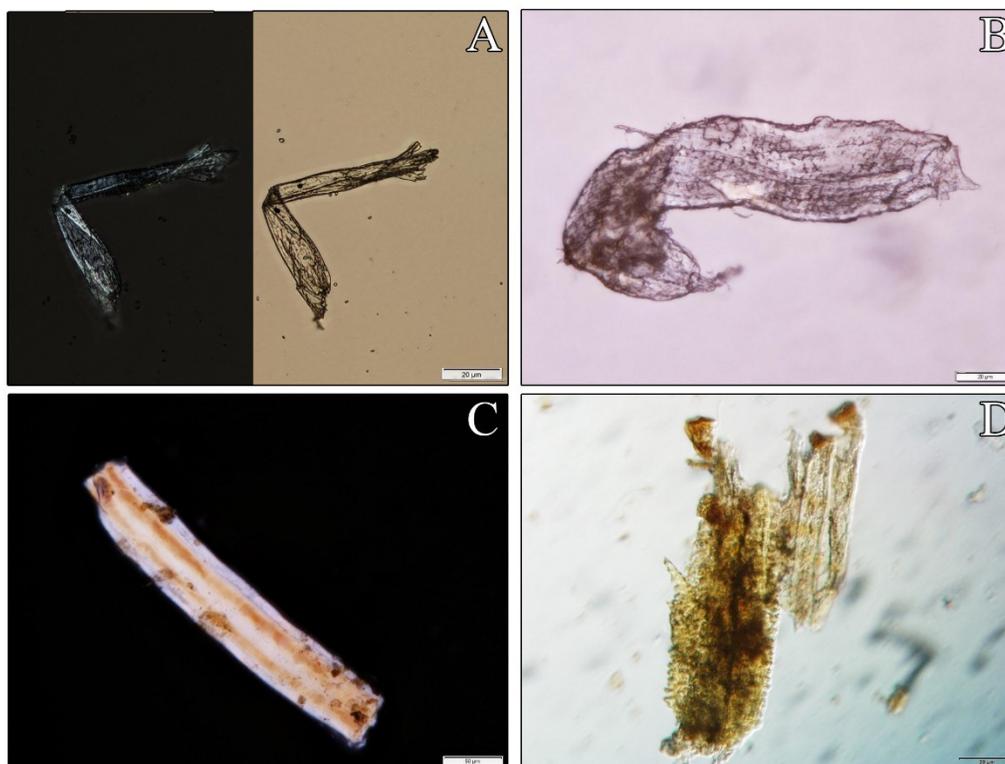


Fig. 7. Examples of plant tissues found within the study sample.

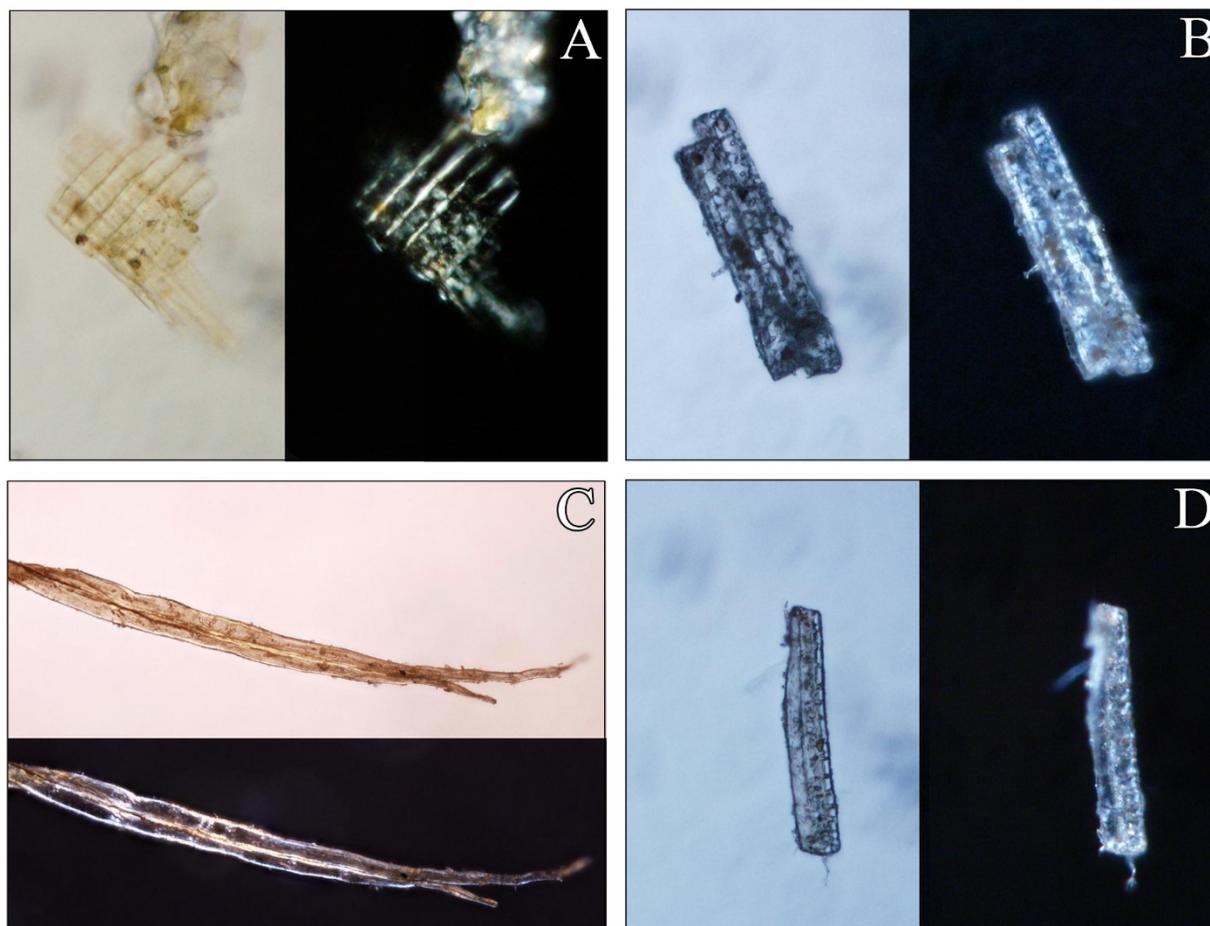


Fig. 8. Examples of woody structural tissues identified within the study sample.

plant and animal. One of the artifacts, UN18, presented a peak consistent with oleanolic acid. This, in combination with several other chemicals that could indicate plant contact (trans-9-hexadecanoic and dodecanoic acid, octadecanoic, nonanoic, and propanoic acids, and dyhydroxanthin) resulted in an extremely strong interpretation (Fig. 13). Weaker results were achieved with nine additional samples, wherein seven were consistent with possible plant contact, and two with combined possible plant and animal contact. Interpretable compounds were not identified in three samples. Table 5 lists the interpreted compounds found within the analysis. For a complete breakdown of identified compounds and retention times, please see Appendix A.

3.2 Interpretive Value

The interpretive value scale was based on the number of results achieved through the use of all five methodologies. Each result counted as a positive score, regardless of inter-methodological consistencies. Biochemical tests and absorbance spectroscopy scales each scored zero to 0.5, incident light residues scored zero to one, transmitted light images scored zero to two, depending on their quantity and interpretability, and GC-MS values scored zero to four, based on the strength of the interpretation, for a total of eight possible points. A total breakdown of interpretative value points can be found in Graphs 1 and 2. The final ranking based on point value resulted in negative, low, medium, high, and very high scores.

Negative interpretive values were not observed within this study. It was found in each sample that a minimum of one method provided positive results. Low values were observed in

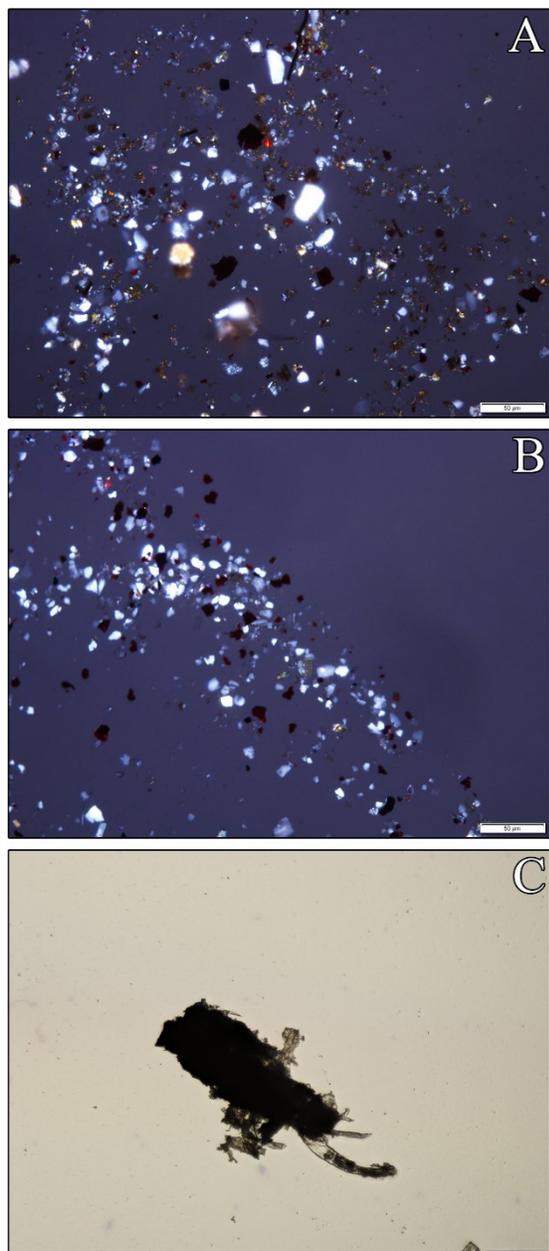


Figure 9: Charcoal, hematite (A,B), and burnt carbon matter (C).

three samples, UN6, UN11, and UN23. In each of the samples, interpretable results were only attained through a single method – biochemical testing for UN6 and UN23, and GC-MS for UN11. Despite the lack of interpretative value, cautionary interpretations of tool use are still possible. Medium values were observed in eight samples. This was the second most common designation, and frequently resulted from positive results in the biochemical testing, transmitted



Fig. 10. Phytoliths (A,B) and a single whisker raphide (C).

light microscopy, and GC-MS interpretations. Results within this ranking provide a stronger interpretation than that listed previously, but require a medium to high ranking within the interpretative strength for a strong overall interpretation (see Section 3.3). High values were

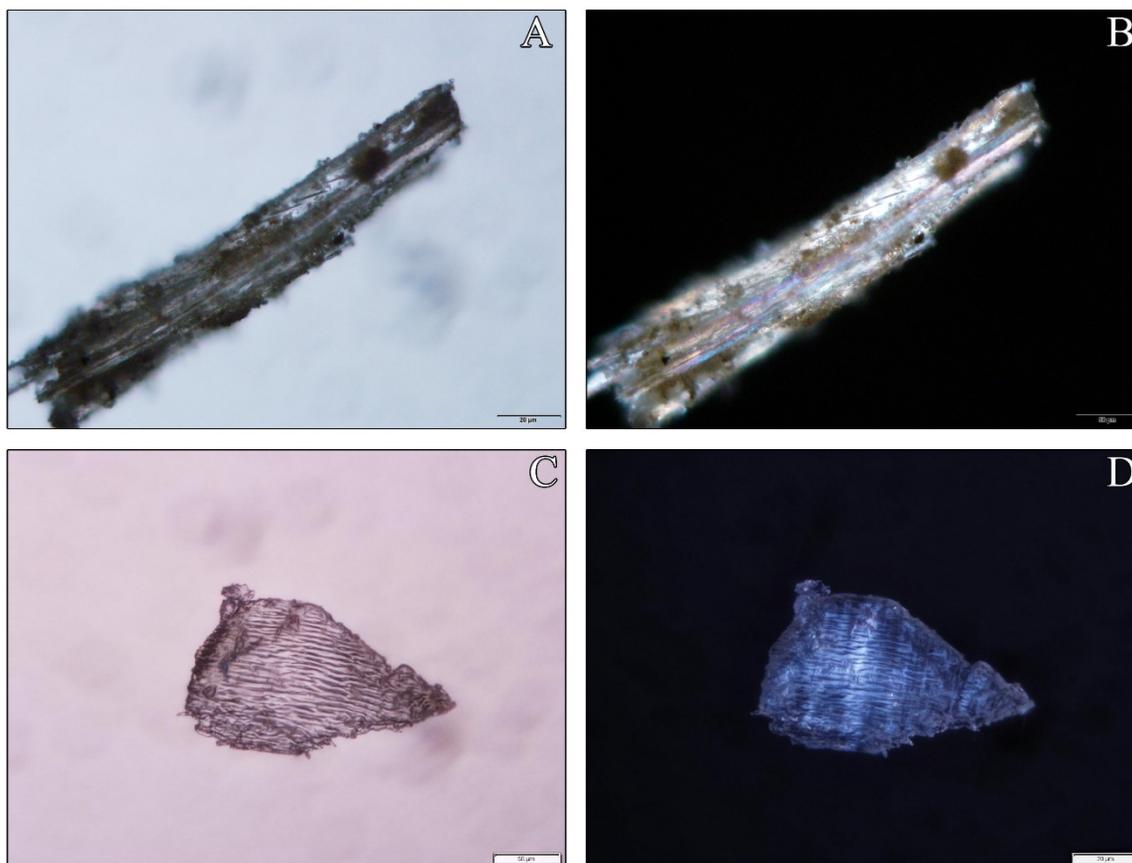


Fig. 11. Highly degraded feather and shell in plane (A,C) and cross-polarized (B,D) light.

observed in eleven samples, making this ranking the most common within the study. This designation resulted from a combination of positive biochemical tests or *in situ* residues being visible in addition to one or more interpretable transmitted light residues, and interpretable GC-MS data. Strong interpretations are possible within this ranking and are supported, but not determined by, the interpretive strength score. Lastly, very high interpretable values were observed in a single artifact within the study. Positive results were attained in each method applied to UN18. Despite the inconsistent data from each method and subsequent low interpretive strength, the quantity of data combined with the diagnostic strength of the GC-MS results provide an in-depth interpretation.

3.3 Interpretive Strength

Interpretive strength was based on inter-methodological consistency. Negative interpretive strength implies that results were attained from more than one methodology, but were indicative of different source materials. Weak positive results existed when results from a single methodology were achieved. Positive results were noted when two to three methodologies provided consistent results, and four or more consistent results allowed for a strong positive strength score. The individual strength determination of each sample can be found in Table 6.

Results with negative interpretive strength were observed in two of the samples, UN19 and UN23. The samples had positive results in two and three methodologies, respectively, but each positive result failed to be consistent with those from alternate methods. Weak positive strengths were observed in a single sample, UN11, in

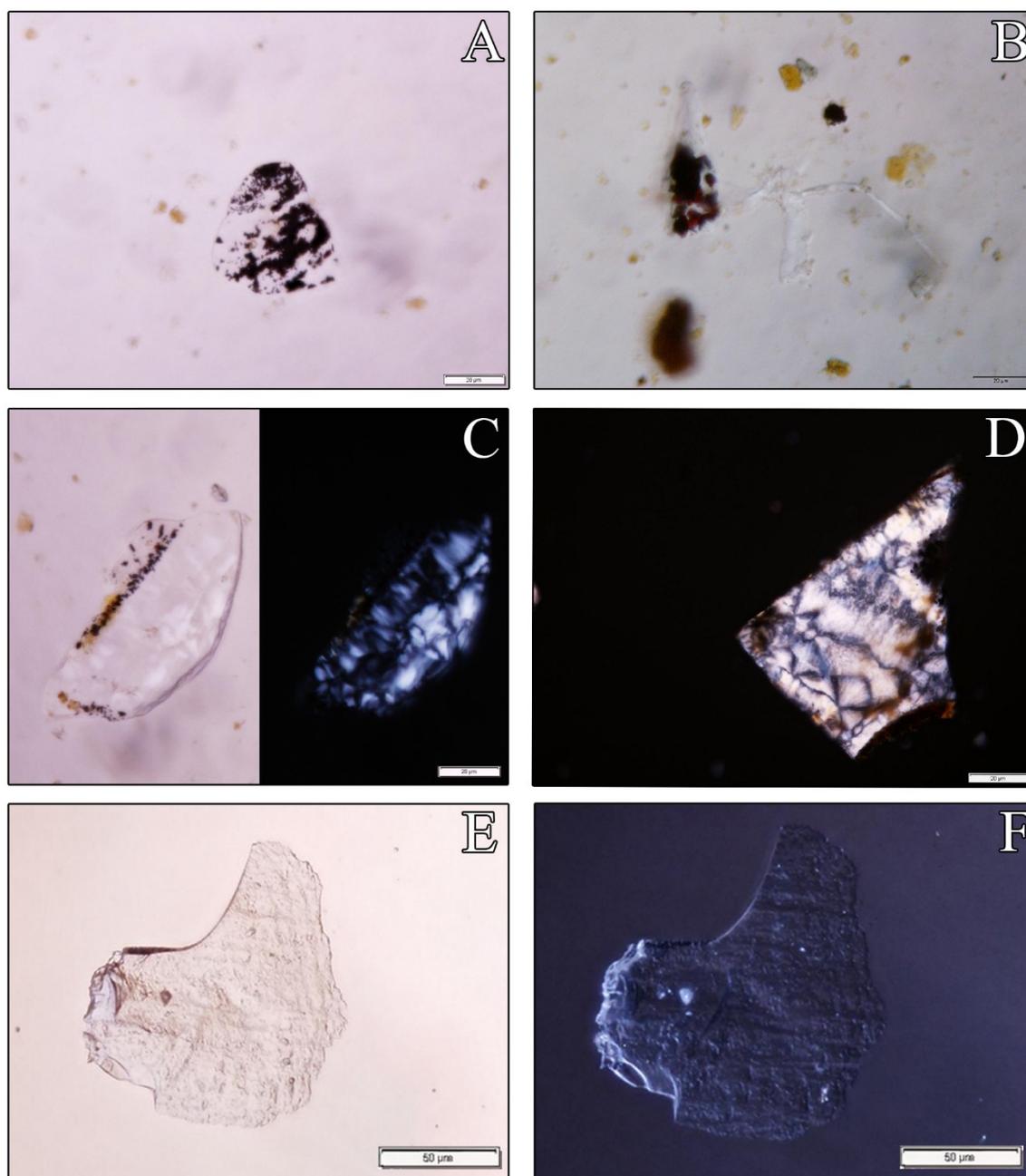


Fig. 12. Microscopic flakes identified in several samples. Materials included from taconite (A,B), chalcedony (C,D), and chert (E,F).

which results were only attainable via GC-MS. Positive strengths were noted in fourteen samples. Successful results were primarily achieved from GC-MS and transmitted light microscopy, with consistencies between the biochemical tests and absorbent light spectroscopy frequently noted through positive

carbohydrate and fatty acid tests. Alternately, consistency with the incident light microscopy proved to be fairly uncommon due to the low frequency of visible residues. Strong positives were noted in UN14, UN18, and UN25, in which each methodology not only tested positively, but maintained inter-methodological consistency.

Each method and the overall strength were tested as independent variables to determine if statistically significant relationships could be found. A Kruskal-Wallis H-test was employed, the results of which are found in Table 7 and are discussed below.

Table 2
Transmitted Light Microscopic Analysis: Condensed Results

Particulate	Number	Reference
taconite microflake	5	Hodgson 2016e
woody tissue	6	Miller 1994; Noyes 2011; Langejens and Lombard 2015; Tobimatsu 2013
plant tissue	10	Michaud 2011; Petraco and Kubic 2004; Chen and Kluver 2010; Horrocks and Lawlor 2006; Ribeiro and Oliveira 2014; Organic Components 2011
charcoal/hematite	5	Organic Components 2011
collagen fibers	3	Fullagar 2006; Stephenson 2015; Langejens and Lombard 2015
chalcedony microflake	2	Frondel 1962; Luedtke 1994; Maggetti and Messiga 2006
Shell	1	Hodgson; Xu, Ying et al. 2015.
cellulosic fiber	2	Petraco and Kubic 2004
feather	1	Robertson 2002; Loy and Nugent 2002
Phytolith	3	Brown 1984; Piperno 2006
whisker raphide	1	Crowther 2009
pigment, plant exudate	1	Petraco and Kubic 2004
chert microflake	1	Hodgson 2016e

4.0 Discussion

Strong results are those which have both a high interpretative strength and value, providing not only significant individual results, but also a high level of inter-methodological consistency. Those with both low strength and value determinations result in limited interpretative power, the results of which must be viewed with caution. Mid-range scores likewise provide tentative results but to a stronger degree than low-range scores.

Using this ranking system, it is possible for a sample interpretation to score high in one system and low in the other. In the case of high strength and low interpretive value, a sample may yield

consistency of results but from fewer lines of evidence. Conversely, a high interpretive value and a low strength implies positive results in several of the methodologies tested, but a lack of consistency exists between those results. The second scenario results in a broader interpretation that must remain speculative due to the lack of consistency between lines of evidence, while the former provides a stronger, yet overall narrower interpretation.

Based on the individual success rates of each of the methods tested, using a range of methodological approaches will not detract from an overall interpretation. The use of multiple lines of evidence present the greatest opportunity of attaining interpretable data, whether or not the final results indicate similar sources. However, for time and cost considerations, it may not prove necessary to complete as many methods as were pursued here. Statistically significant relationships were found between transmitted light microscopy and GC-MS, as well as between both GC-MS and incident light microscopy in relation to the overall interpretative score. Employing both microscopic and chemical analyses appears to provide consistently strong interpretations.

5.0 Conclusion

Results of varying degrees of strength were determined through a broad, multi-analytical approach for each sample examined within the study. Whether weak or strong, some level of interpretation became possible, and statistically significant relationships between methodological consistencies and efficient multi-analytical methods could be determined. This highlights the need for multi-analytical examinations in residue analysis. For example, if biochemical testing and incident light microscopy had been the only two methods employed, fewer results would have been obtainable, resulting in lower overall interpretative strengths and values.

The process described here is especially pertinent for samples recovered from environments that do not typically preserve macroscopic organic remains. Many of these environments are only beginning to be tested for their potential to preserve microscopic organics. In scenarios such as these, multi-analytical analysis becomes increasingly valuable as the

Table 3
Summary of Biochemical Results

	Solani/Sardano - 540nm		Diphenylamide - 595nm		Starch	
<i>Blank</i>	0.157	0.188	0.097	0.092	0.056	0.054
Sample	Test 1	Test 2	Test 1	Test 2	Test 1	Test 2
UN1	0.182	0.165	0.41	<u>0.445</u>	0.052	0.059
UN2	0.111	0.076	0.127	0.133	0.043	0.051
UN3	0.167	0.148	0.08	0.108	0.055	0.062
UN4	0.147	0.162	0.123	0.091	0.062	0.081
UN6	0.187	0.198	<u>0.216</u>	0.096	0.062	0.046
UN8	0.121	0.125	0.126	0.118	0.076	0.088
UN9	0.14	<u>0.203</u>	0.183	0.195	0.049	0.058
UN10	0.126	0.156	0.189	0.154	0.039	0.047
UN11	0.145	0.194	0.195	0.071	0.049	0.082
UN13	0.169	0.139	0.085	0.22	0.058	0.053
UN14	<u>0.216</u>	0.135	0.084	0.071	<u>0.115</u>	0.073
UN15	<u>0.209</u>	<u>0.449</u>	<u>0.341</u>	<u>0.359</u>	0.093	0.076
UN18	<u>0.256</u>	0.175	0.14	0.16	0.049	0.058
UN19	0.076	0.148	0.083	0.086	0.038	0.07
UN21	0.15	0.14	0.102	0.096	0.048	0.084
UN23	<u>0.226</u>	0.15	0.083	0.066	0.059	0.052
UN24	0.177	<u>0.217</u>	0.076	0.088	0.059	0.05
UN25	0.117	0.124	0.078	<u>0.222</u>	0.073	0.094
UN26	0.14	0.144	0.132	<u>0.202</u>	0.051	0.067
UN27	<u>0.212</u>	0.174	0.083	0.121	0.038	0.044
UN29	0.125	0.133	<u>0.268</u>	0.179	0.05	0.062
UN30	0.187	<u>0.313</u>	0.24	0.212	<u>0.476</u>	0.194

survivability of interpretable organic residues is not currently known.

The use of multi-analytical residue analysis within boreal climates, areas not known for organic preservation, needs to be further explored in order to increase the interpretative value of lithic artifacts beyond the roles they have traditionally been ascribed. This study found that the use of five complementary methodological approaches, while time consuming, provides a large amount of data concerning perishable or otherwise invisible resource exploitation within the Early Holocene period in the Northern Superior Region. Due to its time-consuming nature, the process described here would not be suitable for exceedingly large sample sizes. The quality of results, however, demonstrates the importance of implementing additional analyses to small samples recovered from Northwestern Ontario, as it remains one of the few ways to ascertain and quantify evidence indicative of organic resource use.

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Table 4
Individual Analysis - GC/MS Strength Breakdown per Sample

Sample	Interpretive Compounds	Strength	Interpretation
UN1	hexadecanoic acid octadecanoic acid myristic acid azelaic acid	3	Animal
UN2	octadecanoic acid hexadecanoic acid heptadecane tridecane pentadecane azelaic acid	3	Animal
UN3	octadecanoic acid cis-9-octadecanoic acid) trans-9-hexadecanoic acid) myristic acid dodecanoic acid propanoic acid (lactic)	4	Plant (weak animal)
UN4	dioxa-disilaoctaine	1	Tentative plant
UN6	N/A	0	N/A
UN8	propanoic acid octadecanoic acid hexadecanoic acid trans-9-hexadecanoic acid benzene tetradecanoic acid nonanoic acid propanoic acid/lactic	4	Plant/burnt organic
UN9	octadecanoic acid hexadecanoic benzaldehyde pentadecanoic acid hexadecanoic acid	2	Tentative plant and animal
UN10	borate	1	Tentative plant
UN11	octadecanoic acid hexadecanoic acid benzene tetradecanoic acid dodecanoic acid octadecenynoic acid propanoic acid	4	Plant and burnt organic

	glyoxylic acid, di-TMS		
UN13	octadecanoic acid	4	Plant
	palmitelaidic acid		
	benzene		
	tetradecanoic acid		
	dodecanoic acid		
	hexadecanoic acid		
	octanoic acid		
	propanoic acid		
	glyoxylic acid, di-TMS		
UN14	glyoxylic acid, di-TMS	2	Tentative plant (weak animal)
	dodecanoic acid		
	trimethylsilyl ether of glycerol		
UN15	propanoic acid	2	Tentative plant
	dimethylsilyloxytridecane		
UN18	hexadecanoic acid	4	Plant
	oleanolic acid		
	methanone		
	dihydroxanthin		
	octadecanoic acid		
	palmitelaidic acid		
	dodecanoic acid		
	octadecanoic acid		
	propanoic acid		
UN19	glyoxylic acid, di-TMS	1	Tentative plant
UN21	glyoxylic acid, di-TMS	1	Tentative plant
UN23	n/a	0	N/A
UN24	glyoxylic acid, di-TMS	1	Tentative plant
UN25	octadecanol	2	Plant
	dodecanoic acid		
	ethanedioic acid		
UN26	glyoxylic acid, di-TMS	1	Tentative plant
	<i>ethanedioic acid</i>		Tentative plant and animal
UN27	<i>benzene</i>	1	
UN30	N/A	0	N/A
UN33	hexadecanoic acid	4	Plant
	ethanedioic acid		
	gluconic acid		
	octanoic acid		
	dimethyltrioxasilatetradecanol		
	glyoxylic acid, di-TMS		

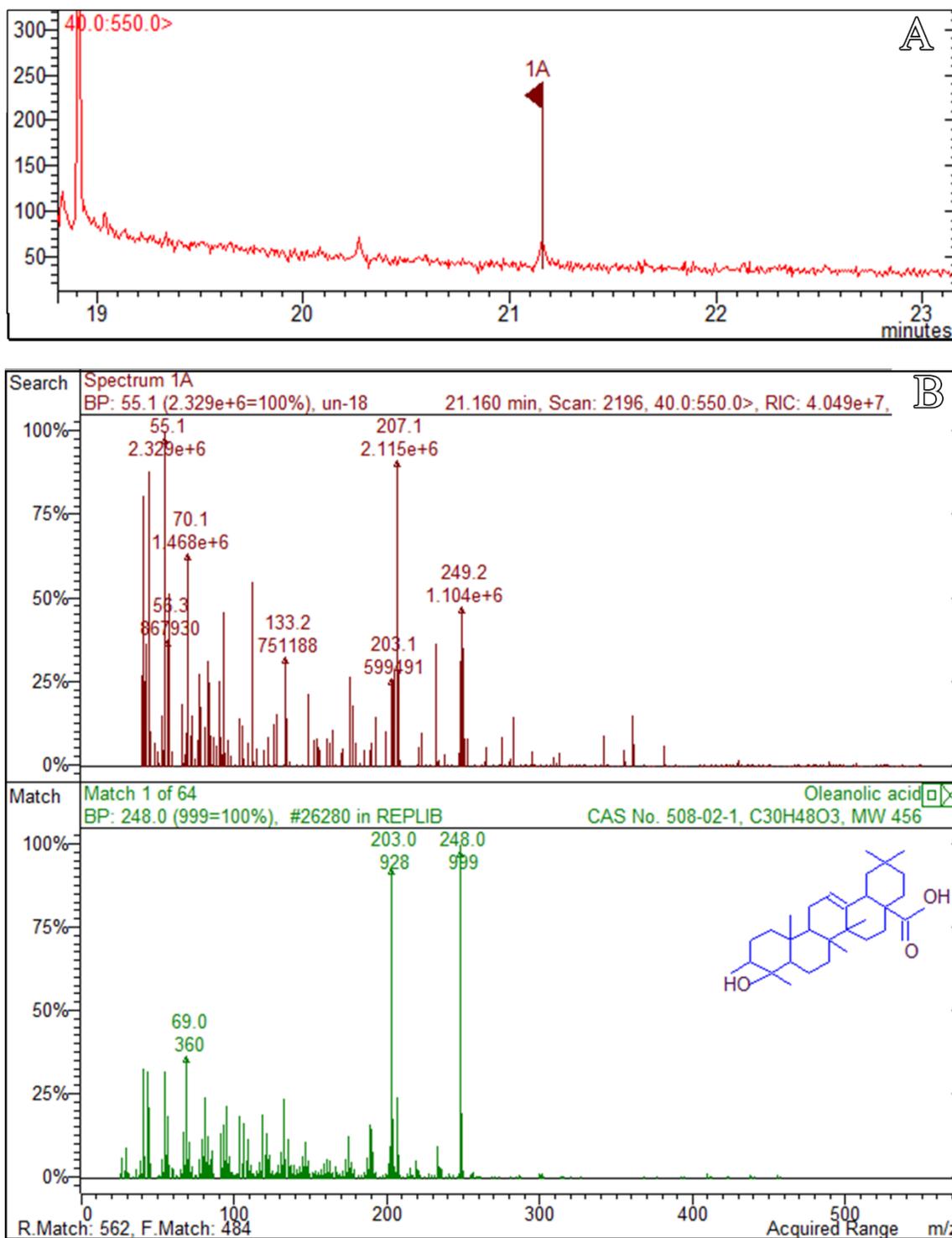


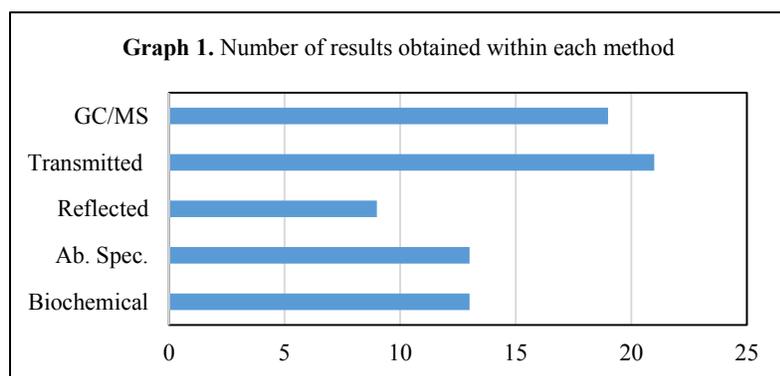
Fig. 13. Total ion chromatogram (A) and the mass spectrum (B) of UN18 at a retention time of 21.160.

Table 5

Summary of compounds included in the GC/MS analysis and interpretation

Compound	Recorded	Possible Sources	Citation
azelaic acid	2	aged oxidation of large fatty acids (rancidity), acne cream, plant, animal	Garelnabi et al 2010; Nicolet and Liddle 1916; Erkins 2002; Al-Shammari et al 2012
benzaldehyde	1	burnt organic material	
Benzene	5	burnt organic material	
carboxylic acid	1	plant	
dihydroxanthin	1	plant, degraded purines	
dimethylaminomethyl, hydroxybenzofuran methanone	1	plant	
dodecanoic acid	8	plant	Chinwe et al 2014
Ethanedioic	4	plant or animal (oxalic acid)	
glyloxalic acid	9	possibly plant	
Heptadecane	1	heptadecanes (17 carbons), burnt plant material, beeswax	Maia and Nunes 2013; Regert et al 2001; Kaal et al 2008; Kaal et al 2009
hexadecanoic acid	9	plant, animal, beeswax, handling, contamination	Malainey et al 1999; Regert et al 2001; Maia and Nunes 2013; Lakshmi et al 2012; Croxton et al 2010; Michalski et al 2013; Prakash et al 2011
trans-9-hexadecanoic acid	4	plant and animal milk	
Icosane	1	icosanes (20 carbons), plant, burnt plant material	Wang et al 2006; Kaal et al 2008
nonanoic acid	9	pelargonic acid - plant, industrial use	Knudsen et al 1993
octadecanoic acid	9	plant, animal, beeswax, handling, contamination	Malainey et al 1999; Regert et al 2001; Maia and Nunes 2013; Lakshmi et al 2012; Croxton et al 2010; Michalski et al 2013
cis-9-octadecanoic acid	1	plant, animal	
trans-0-octadecanoic acid	1	plant, animal milk, fat	
octadecanol	1	plant	
octadecenynoic acid	1	crepenynic acid methyl ester - plant - seeds	
octanoic acid	2	caprylic acid, plant and animal	
oleanolic acid	1	resin exudate, triterpenoid	
Pentadecanol	1	plant	

Pentadecane	1	pentadecanes (15 carbons), burnt plant material	Kaal et al 2008
n-pentadecanoic acid	1	animal fat, milk	
propanoic acid	10	plant residue, seed, nut and/or root	lactic acid
tetradecanoic acid	7	myristic acid, plant <i>calophyllum</i> , plant oils and animal fats	Malarvizhi and Ramakrishnan 2011; Gutiérrez et al 1999; Ertas et al 2014; Azmat et al 2010; Al-Shammari et al 2012; Fievez et al 2011; Maya et al 2006; Gnanamuthu and Rameshkumar 2014; Saravanan et al 2013; Abirami and Rajendran 2011; Sutha et al 2011; Kale et al 2011; Maruthupandian and Mohan 2011b; Ogunlesi et al 2010b
Tridecane	1	tridecanes (13 Carbons), burnt plant material	Kaal et al 2008; Kaal et al 2009
tripropylsilyloxy-undecane	1	plant alcohol	



Graph 2: Interpretive Value Breakdown and Total

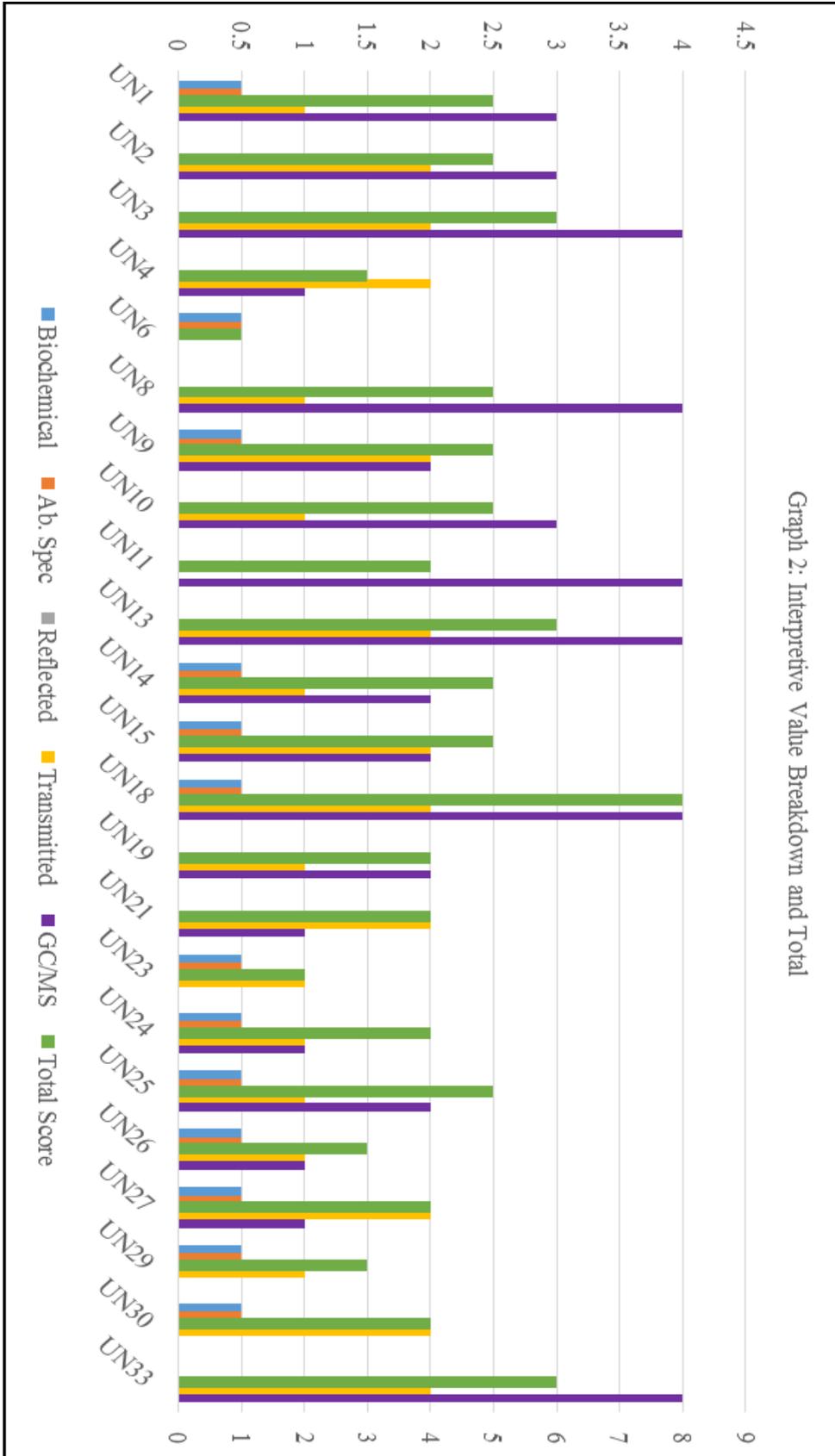


Table 6
Interpretive Strength Breakdown*

Sample	Biochem/ AbSpec	Reflected	Transmitted	GC/MS	Strength (0-4)
UN1	x			x	2
UN2			x	x	2
UN3			x	x	2
UN4			x	x	2
UN6	x				1
UN8			x	x	2
UN9	x		x	x	3
UN10		x	x	x	3
UN11				x	1
UN13			x	x	2
UN14	x	x	x	x	4
UN15	x		x	x	3
UN18	x	x	x	x	4
UN19			x	x	0
UN21		x	x	x	3
UN23	x		x		0
UN24	x	x	x	x	4
UN25	x	x	x	x	4
UN26	x		x	x	3
UN27	x		x	x	3
UN29	x	x	x		3
UN30	x	x	x		3
UN33			x	x	2

*Consistent methodologies are marked with an 'x.'

Table 7
Kruskall-Wallis H-Statistic Values

Test Variable (Dependent)	Group Variable (Independent)			
	Bio/AbSpec	Reflected	Transmitted	GC/MS
Bio/AbSpec	X	0.202	0.242	0.059
Reflected	0.202	X	0.787	0.492
Transmitted	0.242	0.787	X	0.005
GC/MS	0.059	0.492	0.005	X
Strength	0.164	0.004	0.135	0.069

Note: significant values are marked in bold

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CHAPTER FIVE

EARLY HOLOCENE SUBSISTENCE VARIABILITY WITHIN NORTHWESTERN ONTARIO: INCORPORATING LITHIC USE-WEAR AND RESIDUE ANALYSIS FOR THE DETECTION OF PERISHABLE TECHNOLOGIES

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CHAPTER INTRODUCTION

This chapter serves as the discussion and conclusion of the thesis. Results from both broad methodological approaches are synthesized and discussed together for the first time, allowing detailed interpretations of tool function and source materials supported by several independent lines of evidence. Based on these results, a discussion of technological variability among unifacially flaked artifacts and resource exploitation at WPII is presented. For comparative purposes, all tool metrics were recorded and are presented in Appendix D. A table summarizing all results is located in Appendix E.

The sample size of this study is small relative to those common with other analytical approaches (i.e. reduction sequence or spatial analyses). Due to its size, it cannot be used to characterize all Early Holocene sites within Northwestern Ontario, and perhaps not even to characterize the intra-assemblage variability of the Woodpecker II site. Rather, this thesis illustrates ‘proof of concept’ of micro-analytic approaches to archaeological assemblages recovered from challenging depositional environments. Despite this limitation, the level of detail in the results achieved throughout the case studies presented here clearly demonstrate the effectiveness of the techniques toward site specific interpretation. This is of particular relevance to Early Holocene assemblages with little to no organic component.

The results herein demonstrate that the strict dichotomy between Paleo and Archaic cultural horizons and their associated technological and resource traditions overlooks complexities possible in the long transitional period. A varied unifacial tool kit is indicated: the small sample consisted of artifacts with both generalized and specialized functions including scraping, planing, and cutting. Generalized, multi-purpose function was indicated on all minimally shaped expedient artifacts. Informal artifacts however, those with intentional shaping, demonstrated a mixture of generalized and specialized function, as well as an unexpected degree of hafting in varying styles. Formal artifacts, although few in number, showed greater tendency toward more specialized use. Organic evidence from faunal, floral, avian, and aquatic resources were identified. Among the unifacial tools, a heavy reliance on single resource types was not indicated. An argument toward the increased use of micro-analytical techniques within Northwestern Ontario is presented as a method to build an increasingly detailed record of changing lifeways of early cultural groups.

**EARLY HOLOCENE SUBSISTENCE VARIABILITY WITHIN NORTHWESTERN ONTARIO:
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OF PERISHABLE TECHNOLOGIES**

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EARLY HOLOCENE SUBSISTENCE VARIABILITY WITHIN NORTHWESTERN ONTARIO: INCORPORATING LITHIC USE-WEAR AND RESIDUE ANALYSIS FOR THE DETECTION OF PERISHABLE TECHNOLOGIES

Tasha Hodgson and Carney Matheson

Abstract: Use-wear and multi-analytical residue analyses were used to analyze formal and expedient Early Holocene artifacts from the Upper Great Lakes of Canada. Podzolic soils of the boreal forest commonly prevent the preservation of organic artifacts, resulting in a need for microscopic methods of detection. Results indicate that the majority of expedient tools were used on a broad variety of resources and that exploitation at Electric Woodpecker II (DdJf-12) included plant, animal, avian, and aquatic sources. The edge damage exhibited demonstrates both generalized and specialized tool use and a large number of composite tools, nearly half of which consisted of informal artifacts. Broader applications of the techniques will aid in the documentation of resource adaptation and subsistence use on a regional scale.

Terrestrial, big-game hunting is consistently offered as the primary interpretative model of subsistence by Early Holocene (10,000 to 8,000 BP) cultural groups from east of the Rockies to the Upper Great Lakes region of Canada (Julig 2002; Kuehn 2007; Mason 1981). Similarly, lithic technologies within the Upper Great Lakes are believed to have changed little throughout the Late Pleistocene to Early Holocene time periods. Limited observable technological change coupled with minimal organic artifacts has resulted in persistent theories of big-game predation regardless of region or environment. Although broader resource bases (Fiedel 1987; Kuehn 2007) and littoral adaptive strategies (Julig 2002) have been proposed, the lack of direct material evidence constrains independent verification. Indirect evidence of these materials may become obtainable through the use of micro-analytical techniques both locally and within other regions (Cesaro and Lemorini 2012; Miller 2014; Newman and Julig 1989).

Early Holocene sites within Northwestern Ontario have been poorly documented because of physical limitations on site discovery, a lack of diagnostic artifacts, poor organic preservation, slow rates of soil deposition, and frequent natural disturbances (Hinshelwood 2004; Julig 1994; Norris 2012; Pilon and Dala Bona 2004). The latter two factors result in little stratigraphic separation between assemblages or occupation events (Hinshelwood 2004). The vulnerability of organic artifacts to bacteria from biologically active soil layers combined with the degradative properties of podzolic soil conditions often prevent the preservation of organic materials common in other regions of Canada (Jennings 1989). Archaeological interpretation of these sites is thus confined to lithic analyses via macromorphic or spatial analyses, techniques which are inherently limited in their ability to assess complexities within Early Holocene subsistence strategies and resource exploitation through the detection of small scale dietary variations.

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Introducing increasingly accurate micro-analytical techniques will expand the knowledge of regional subsistence and provide the quantitative evidence necessary to reconstruct and validate the early Pre-Contact subsistence model practiced within the Upper Great Lakes region. If applied across varying geographical and temporal regions, these techniques are equipped to record localized adaptations to the changing climatic conditions and dynamic landscapes throughout the earliest inhabitable period in Northwestern Ontario.

A multi-analytical approach utilizing both use-wear and residue analyses was completed on a selection of artifacts recovered from the Electric Woodpecker II site (WP11, DdJf-12) near Thunder Bay, Ontario, Canada. A radiocarbon date of 8680 \pm 50 BP (9760-9540 cal BP, 2 sigma, Beta 323410) was obtained from a small charcoal fragment excavated at occupation depth (Norris 2012). The assemblage consists of a varied toolkit of projectile points, drills, adzes, unifacial and bifacial knives, scrapers, planers, and graters. Preforms and blanks were frequent, as well as a multitude of debitage from all stages of tool manufacture. Unifacially flaked artifacts exhibited considerable morphological variation and were subsequently selected to form the basis of this study. These unifacially flaked artifacts included both formal and expedient tools. Expedient tools were further divided into informal (intentionally shaped) and expedient (minimally shaped) categories. The micro-analytical techniques described suggest use to enable broad resource exploitation. In addition to this a variety of tool functions and compositions were observed, with nearly half the artifacts bearing damages consistent with hafting.

Site and Environmental Context

The oldest currently dated human occupation along the north shore of Lake Superior is believed to have occurred approximately 9500 B.P. following the final northward retreat of the Laurentide Ice Sheet (LIS). Site location and land use during the Late Pleistocene to Early Holocene were heavily

influenced by geographic location, post-glacial lake sequences, and both local and regional deglaciation (Lowell et. al 2009); see SI). These conditions affected ecological recovery and available biomass, and therefore were critical in site selection by the earliest occupants. Shoreline locations may have been favored by Early Holocene groups due to the high seasonal biological productivity which allowed for a broader, seasonally changing resource base (Fox 1980; Julig 2002).

Electric Woodpecker II (WP11; DdJf-12) is one of a series of sites distributed along the relict shoreline of Glacial Lake Minong approximately 25 km east of Thunder Bay, Ontario, Canada. The sites are collectively characterized by frequent occupation of raised strandlines, the almost exclusive exploitation of Gunflint Formation lithic materials, application of parallel oblique flaking techniques, and varied lanceolate point traditions (Norris 2012). These sites are interpreted as belonging to the Lakehead Complex (Fox 1975, 1980) local expression of the broader Interlakes Composite (Ross 1997).

The study site is situated along a section of shoreline dating to the Glacial Lake Minong period of Lake Superior (see S1). Geoarchaeological interpretations suggest that around the time of occupation the site would have been situated along a portion of storm beach overlooking a shallow backbeach (Norris 2012). Local vegetation consisted of closed spruce forest transitioning into spruce-pine boreal forest. Tree species included jack pine, spruce, and balsam fir, and a northern lichen woodland forest was located less than 50 km to the north (Julig, McAndrews and Mahaney 1990; Julig and McAndrews 1993). The dynamic ecological changes that occurred between 10,500 and 7500 BP would likely have required technological diversification and seasonal shifts in resource exploitation (Anderson and Lewis 1991; Kuehn 2007).

The WP11 site suffers from common regional taphonomic challenges, resulting in the nearly complete absence of organic preservation. This absence of floral and faunal materials has meant that interpretations regarding resource exploitation have largely been left to conjecture utilizing datasets obtained from sites in adjacent regions.

Results

The objective of a multi-analytical approach utilizing both use-wear and residue analyses was to test the replicability of interpretations regarding tool function, including the manner of use, contact material characterization by hardness, and contact material source. As illustrated in Table 1, consistent interpretations were achieved for 13 of the 23 samples. Seven additional samples returned divergent interpretations, while the remaining two samples returned incomplete results.

The presence of conflicting functional interpretation in the sample does not necessarily indicate a failure of one of the methods. Rather, it demonstrates the limitations inherent when analyzing mixed, unknown residues. In six of the samples, the lack of consistency stemmed from edge damages indicating use on fresh meat and bone combinations while the residue analysis indicated compounds consistent with plant use. These conflicting results may indicate that these samples served multiple purposes. Evershed and Tuross (1996) suggest that the greater susceptibility of proteins and amino acids to degradation decrease the likelihood of straightforward identification of such residue. Additional reactive experimental testing is required to more accurately determine this trend. Residue interpretations were unsuccessful in the two remaining samples. Multiple resource types and functional motions were indicated within the sample group (see Table 1).

Residues attributed to plant, animal, aquatic, and avian sources were identified by the presence of cellular components identified within the residues (Fig. 1), in addition to GC-MS chromatograms and biochemical colorometric testing (11). In some cases, the artifact function inferred from the residue and use wear analyses was consistent with that hypothesized based on morphology. For example, this was the case with each of the formalized endscrapers and their use on both dry and wet hides. The function of other unifaces based upon their morphology (i.e.

preforms, drills, thumb and sidescrapers), were sometimes inconsistent with the functions indicated by the macro-analysis. Instead, the analysis use as wood or bone planers, plant knives, or multi-purpose expedient cutting tools. Further functional analysis indicated both unidirectional and bidirectional scraping and planing, unidirectional whittling, and both unidirectional and bidirectional transverse cutting. Source materials identified through use-wear analysis included fresh and dried bone, dry and fresh hide, wood, and both soft and woody plants. Hafting was indicated on nearly half of the samples (both formal and informal artifacts) of varying lithic quality.

In total, four formal and eight informal tools, bore evidence consistent with hafting using one of three techniques: male hafting, in which the hafted portion of the tool was inserted into the shaft (formal=2, informal=6); juxtaposed hafting in which the distal portion of the tool was set upon a shaped portion of the haft and kept in place with hafting adhesive or fibers (formal=2; informal=1); and wrapping, where material is tied over the handheld portion of the tool (informal=1). Expedient artifacts, those with minimal shaping, did not show evidence of hafting. The function of hafted tools include cutting (unidirectional and bidirectional, transverse), planing (unidirectional and bidirectional, longitudinal, pushing), and scraping (unidirectional and longitudinal, pulling). Tools exhibiting these uses included several scrapers (n=6), a knife (n=1), planers (n=4), and a possible graver (n=1). Multi-use was detected in seven hafted artifacts, with common combinations of wood and bone as well as plant and bone. Discrete function was identified on four artifacts: one dry hide scraper (Fig. 2), one fresh hide scraper, one bone planer, and a single knife (with traces of hafting residue) used for butchering. The final hafted artifact, UN33, lacked a working edge due to breakage prior to excavation. While this limited the interpretation concerning its function, the remaining area demonstrated damages consistent with hafting.

The unhafted artifacts consisted of four informal, five expedient, and a single unknown formal artifact. The formal tool edge was included here out of necessity due to the

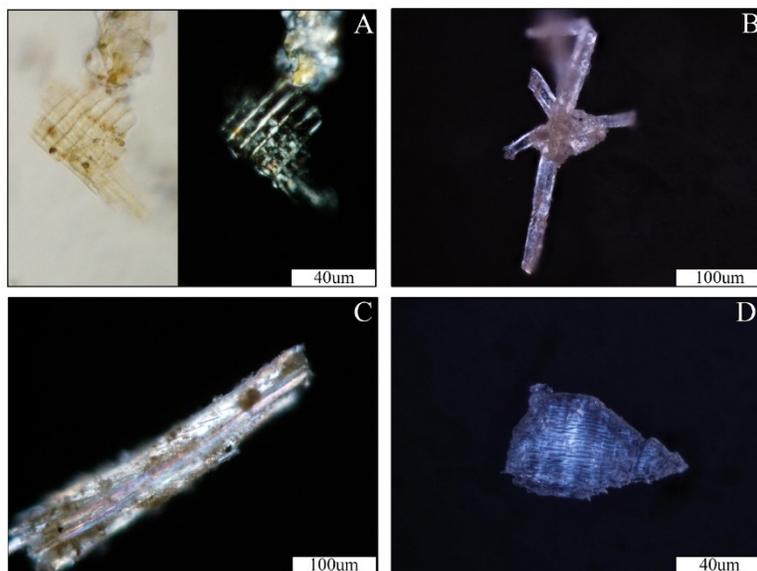


Figure 2. Examples of residues observed throughout the study with 200x to 500x magnification: woody cell tissue (A, 200x), degraded collagenous material likely from an animal source (B, 500x); highly degraded feather barbule fragment (C, 500x); and a small fragment of shell (D, 200x). All images excluded the first half of composite image A were taken under cross-polarized light.

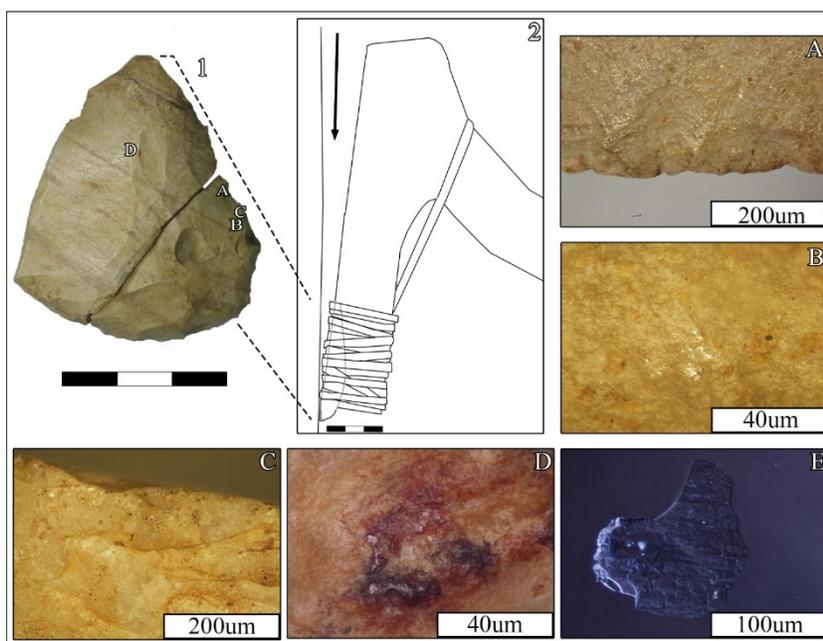


Figure 3. Formal artifact UN30 (1), hafted in a juxtaposed manner (2, modified from Rots 2010). Edge damages and polishes are consistent with use as a dry hide scraper: dull polish evenly distributed across wide margin of tool surface interrupted by scars created shortly before disuse (A, 30x); dull, greasy polish evenly distributed across surface and marked with smaller bright spots (B, 200x); slightly rounded retouch scars on the dorsal surface (C, 100x); probable hafting residue with embedded striations (D, 500x); and a single scalar microflake with edge scarring and perpendicular striations recovered from the residue (E, 500x).

unrecovered distal portion of the tool. Functions represented within this category include planing (bidirectional), scraping (unidirectional and bidirectional), and cutting (bidirectional and unidirectional). Unhafted functional identifications include a single concave planer or spokeshave (n=1; Fig. 3), knives (n=6), and scrapers (n=3). Multi-use was detected in six artifacts with combinations consisting of soft and woody plant, bone and plant, wood and bone, and wood, plant, and bone. Discrete functions were found on the four remaining artifacts, the single formal tool and three informal artifacts. Results indicated use on charred woody material (planer), soft plant material (knife), fresh hide (formal scraper), and bone (spokeshave). A summary of results can be found in Table 1.

Discussion

The strict dichotomy between Paleo and Archaic cultural horizons and their associated technological and resource traditions overlooks the long transitional period and the complexities and inter-mixing of technologies and resources that likely happened therein. Medium to large projectile points have been used to propose continued reliance on big-game predation by Early Holocene people. While arguments supporting a broader, more generalized economy within the region are becoming more common, substantive organic evidence remains elusive (Julig 2002; Kuehn 1998). Use-wear and residue analysis provide an indirect route to continue testing these hypotheses.

While the sample is small in scale and only addresses unifacial tool function, it indicates that resource exploitation at the WPII site was broader than widely thought. Increased application of micro-analytical techniques at sites such as this may aid in the documentation of otherwise invisible resource use, allowing the detection of variable technological and resource exploitation models at local and regional levels. The interpretation indicates that the unifacial tool kit is morphologically more varied than is usually thought. This is reflected in the frequency of informal ‘scrapers’ utilized as cutting and planing

tools, the frequency of minimally shaped expedient artifacts as multi-purpose tools, and the frequency of hafted informal artifacts. This unexpectedly high frequency of hafted informal tools implies that the expediently manufactured artifacts played a more important technological role than standard analyses imply. If employed across a wider regional basis, the applicability of these techniques may aid in the documentation of complexities such as these in Early Holocene occupations across Northwestern Ontario.

Multiple source materials indicated by both use-wear and residue analyses indicate that these tools were used for multiple purposes on diverse substances. While the scope of this project did not permit identification beyond class level, the presence of multiple sources indicates a broader resource base represented within the lithic unifacial artifacts from WPII. The analysis of use-related functions and residues of non-projectile artifact types allows for a broader view of resource exploitation not typically pursued within previous subsistence models (Odell 2003). Complex resource exploitation becomes increasingly visible with multi-analytical techniques, providing the organic component so frequently lacking within Northwestern Ontario assemblages.

The unifacial toolkit recovered from the site is variable in morphology as well as in function and composition. The majority of hafted tools were used in a longitudinal motion; i.e., scraping or planing, while the unhafted artifacts were used equally in both longitudinal and transverse motions, indicating more generalized functions. Both categories displayed discrete wear in equal proportions with use limited to bone and dry hide (hafted) and bone, plant, and woody materials (unhafted). Artifacts with wear indicative of use with dry hide were consistently made of the highest quality materials within the sample. Excluding this trend, it does not appear that formal artifacts were used more extensively or in a more specialized capacity than informal artifacts. It has been suggested that this pattern indicates a hitherto undocumented reliance on expedient technologies within the Upper Great Lakes region during the Early Holocene period (Bamforth 1986; Kelly and Todd 1988).

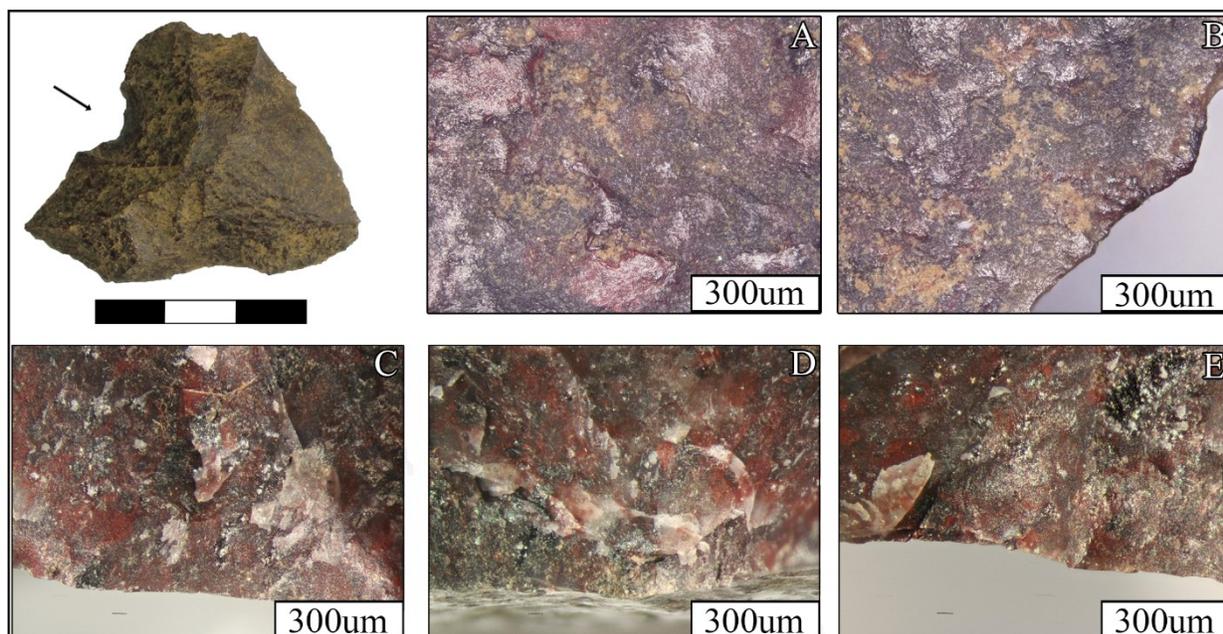


Figure 4: UN1, identified as a bone planer or spokeshave. Working edge is indicated by black arrow. Dull, patchy polish present on smoothed lower surfaces with heavier glossy present on peaks (A, 500x). Polishes are present immediately along working edge and further into the tool surface, with a discontinuous area in between, characteristic of use with use on bone (B, 200x). Lower, middle, and upper portions of concavity showing primarily triangular hinge (n=12) and scalar hinge (n=2) scars, with smaller amounts of scalar stepped and feathered (C, D, E, 30x). Minor nibbling, rounding, and polishing were present as well.

The classes of tools observed in the study include formal, informal, and expedient. Informal and expedient artifacts are frequently overlooked or misidentified as preforms (Odell 2003). The case is made here that this class of tools is equally and perhaps more broadly informative than the more easily recognizable formal artifacts. The informal class appears to have similar functions to formal class artifacts, display wear consistent with hafting, and bear evidence of equally heavy utilization. The continued disregard of expedient artifacts in terms of recoverable data hinders the analysis and interpretation of archaeological sites lacking macroscopic organics. The inclusion of these tools in combination with micro-analytical techniques has the ability to significantly increase the documentation of organic materials from Early Holocene sites.

The use of micro-analytical techniques can inform our knowledge of site-specific resource exploitation in Northwestern Ontario, and broader use of these methods will allow for the documentation of this resource exploitation

and adaptation throughout the dynamic changing landscapes of the Early Holocene period.

Conclusion

Functional interpretations of Early Holocene unifacial artifacts were determined through use-wear and residue analyses. Results demonstrate that holistic approaches to technological analyses are needed to ensure a maximum recovery of data, particularly within regions with poor organic preservation. The use of micro-analytical techniques provides a solution for the lack of interpretable organic materials. Results further indicate site-specific utilization of avian, aquatic, faunal, and floral resources at WP11, and demonstrate that the early occupants of the site exploited a wide range of resources and manufactured a variety of tools with both specialized and generalized functions. Additional and regionally varied use of micro-

TABLE 1**Summary of synthesized results from the use-wear and residue study of unifacial tool types at WPII.**

ID	Type	Hafted	Motion	Direction	Contact Materials	Residues	Final Interpretation
UN1	Expedient	No	Push-Pull	Bidirectional, Longitudinal	Medium; fresh wood, fresh bone	Animal fats, harder materials (microflake): bone likely	Handheld bone spokeshave/planer
UN2	Expedient	No	Cutting	Bidirectional, Transverse	Soft plant, woody plant	Charred woody plant material; small amount of animal	Multi-purpose handheld knife
UN3	Informal	M	Pull	Unidirectional, Longitudinal	Soft plant	Plant roots and fatty animal materials	Multi-purpose hafted scraper, root and animal processing
UN4	Informal	M	Push-Pull	Bidirectional, Longitudinal	Medium to hard; seasoned wood and bone	Wood materials, charred.	Hafted wood planer
UN6	Informal	J	Push-Pull	Unidirectional, Longitudinal	Dry hide	No residue interpretation possible	Hafted dry hide scraper
UN8	Informal	No	Cutting	Bidirectional, Transverse	Medium-hard; dry wood	Plant, woody plant, or wood	Handheld expedient knife
UN9	Informal	M/J	Pull	Unidirectional, Longitudinal	Medium-hard; dry wood	Plant or animal, burnt organics	Hafted wood planer
UN10	Formal	M	Push-Pull	Bidirectional, Longitudinal	Medium; fresh bone	Weak plant, possible animal	Hafted bone planer
UN11	Expedient	No	Cutting	Bidirectional, Transverse	Soft and medium; fresh bone, meat	Plant materials, seeds, burnt plant	Multi-purpose expedient knife
UN13	Informal	No	Pull	Unidirectional, Longitudinal	Medium, medium-hard; fresh bone or wood	Plant, woody plant, charred plant possible	Multi-purpose handheld scraper
UN14	Informal	M	Pull	Unidirectional, Longitudinal	Soft and medium-hard; fresh bone, meat	Weak plant, possibly contamination	Multi-purpose hafted scraper
UN15	Expedient	No	Pull-cut	Unidirectional, Transverse	Soft and medium-hard; fresh bone, meat	Structural plant material	Multi-purpose handheld knife
UN18	Informal	M	Cutting	Bidirectional, Transverse	Soft and medium-hard; fresh bone, meat	Plant, plant resin	Hafted knife, butchering
UN19	Informal	M	Pull	Unidirectional, Longitudinal	Soft; fresh hide, soft plant	Plant and feather	Multi-purpose hafted scraper
UN21	Formal	M	Pull-cut	Unidirectional, Transverse	Medium, medium-hard; dry bone and wood	Plant; fibrous material	Hafted whittler; used on wood, fibrous plant materials, or bone.
UN23	Formal	N/A	Pull	Unidirectional, Longitudinal	Fresh hide	Very weak plant and animal	Fresh hide scraper
UN24	Informal	W	Pull	Unidirectional, Longitudinal	Medium-hard; dry wood, bone	Structural plant use; hafting residue not picked up by GC/MS	Wrapped wood and woody plant scraper

UN25	Informal	No	Pull-cut	Unidirectional, Transverse	Soft; plant and meat	Tentative plant	Handheld general purpose knife
UN26	Informal	No	Pull-cut	Unidirectional, Transverse	Medium-hard; dry wood, dry bone	Structural plant material, possible outer covering	Handheld general purpose knife
UN27	Expedient	No	Push	Unidirectional, Longitudinal	Medium-hard; dry bone, wood	Woody plant or wood, possibly charred	Handheld wood planer or scraper
UN30	Formal	J	Push	Unidirectional, Longitudinal	Dry hide	Probable hafting residue visible, undetected by GC/MS	Hafted dry hide scraper
UN33	Formal	J	N/A	N/A	Abrasive wear on arrises and both lateral edges consistent with hafting.	No interpretable residues.	Inconclusive due to missing working edge; possible adze or chopper

*M=Male; J=Juxtaposed; W=Wrapped

analytical studies may allow for an increasingly detailed record of changing lifeways of early Northwestern Ontario cultural groups.

Methods and Materials

Samples

Twenty-two unifacially flaked artifacts were subjected to sequential use-wear and residue analyses. The sample was morphologically highly variable, consisting of unifacially flaked tools of various levels of manufacture and field identified as scrapers, sidescrapers, endscrapers, preforms, drills, and flakes. Artifacts were stored individually in plastic bags from the time of excavation, and powder-free nitrile gloves were worn during artifact handling to limit exposure to modern contaminants. Working edges were first identified with the use of a hand lens (16x). *In situ*, partially matrix covered residues were photomicrographed using a Nikon SMZ864 stereoscopic microscope with a Canon EOS70D DSLR camera and Varian II adapter (1.5x). Primarily 30x magnification was used, with increases up to 97x as necessary. The selected edges were then sonicated for 45 minutes in sterile, acid-washed glass vessels. Edge analysis was then completed prior to any further residue

analysis. Detailed descriptions of the methodological protocols can be found in Hodgson 2016a and Hodgson 2016b.

Use-wear Analysis

The working edges and/or hafting areas were submerged in a solvent solution containing equal parts acetonitrile, ethanol, and double-distilled water. The samples were sonicated for 45 minutes and allowed to air-dry. Artifacts were then examined using a Nikon SMZ864 stereoscopic microscope with a Canon EOS70D DSLR camera and Varian II adapter (1.5x). Magnifications varied from 30x to 97x. Multiple images were taken every two to four millimeters along the working edges and stacked with ZereneStacker© image software to ensure adequate depth of field. Flake scar and feature counts were recorded in three to five locations evenly distributed across the tool edge to provide an overview of edge wear. Further analysis of edge and surface wear was completed with transmitted light microscopy using an Olympus BX51 microscope with magnifications of 100x to 500x. Locations of observed wears were recorded on overview photographs of each artifact. Wear patterns were then compared to those from an experimental database as well as to those within current literature (Odell 2003; Rots 2010).

Residue Analysis

The cleaned edges of each sample were re-examined for *in situ* residues with a high-powered incident light microscope (100x, 200x, 500x); residues were then recorded on overview photographs. Portions of the extracted residues were used for colorimetric biochemical characterization, as well as desiccated and mounted for transmitted light microscopic analysis. A larger portion of each was then freeze dried, derivatized, and submitted for gas chromatography coupled mass spectroscopy (GC-MS). Each method was analyzed individually; interpretations for each artifact were made through a synthesis of individual results.

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Data Availability Statement

The preceding manuscript is one of four articles that were included in the author's Master's thesis through the Lakehead University Master's of Environmental Studies – Northern Environments and Cultures (MES-NECU) program. The thesis in entirety and all the associated data can be accessed through the Lakehead University Knowledge Commons – Electronic Theses and Dissertations web page (<http://lurepository.lakeheadu.ca/handle/2453/5>). Hard-copies of the thesis can be requested through Library Services or from the author.

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SUPPORTING TEXT AND IMAGES

SUPPORTING TEXT

Site location and use during the Late Pleistocene to Early Holocene is believed to have been heavily influenced by geographic location, post-glacial lake sequences, and both local and regional deglaciation rates and topographies (Larson and Schaetzl 2001). The Laurentide Ice Sheet (LIS) began its last major retreat into the Great Lakes Watershed between 21,000 to 18,000 C¹⁴ yr BP (Dyke 2003), with several smaller periods of retreat and readvance over the next several thousand years (Larson and Schaetzl 2001). Regional deglaciation within Northwestern Ontario occurred between 12,000-10,000 C¹⁴ yr B.P. (Lowell et al 2009). The final retreat was marked by fluctuating water levels and resulted in several moraines permanently marking the landscape. The Thunder Bay region became habitable no later than 9380 +/- 150 (Zoltai 1965; Julig et al 1990). While it has been suggested that the Upper Great Lakes region may have supported human occupation prior to the Marquette re-advance (~10,024 C¹⁴ yr B.P.); any evidence of occupation has been removed by the encroaching ice sheet (Philips and Hill 2004). High sandy ridges created throughout the deglaciation process provided ideal locations for temporary or long-term occupation. The proximity to freshwater for both travel and sustenance in addition to the higher biodiversity associated with such regions would have made such locations very attractive to past populations (Kuehn 1998; Julig 2002).

Shoreline locations may have been favored by Early Holocene groups due to the high biological productivity of the area which allowed for a broader, seasonally changing resource base (Fox 1976; Julig 2002). This trend seems to be particularly strong in shoreline locations with easy access to lithic materials. Beaver, bison, moose, caribou, and fish have been reported across the Great Lake Region as valuable resources for Plano peoples (Julig and McAndrews 1993). The coastal orientation implies the importance of aquatic resources like beaver, fish, and waterfowl (Julig and McAndrews 1993; Kuehn 1998). Material evidence of pre-ceramic resource use does not typically exist due to the poor preservation of the area, yet general foraging economies within northern circumpolar regions are common within the ethnographic data (Julig 2002).

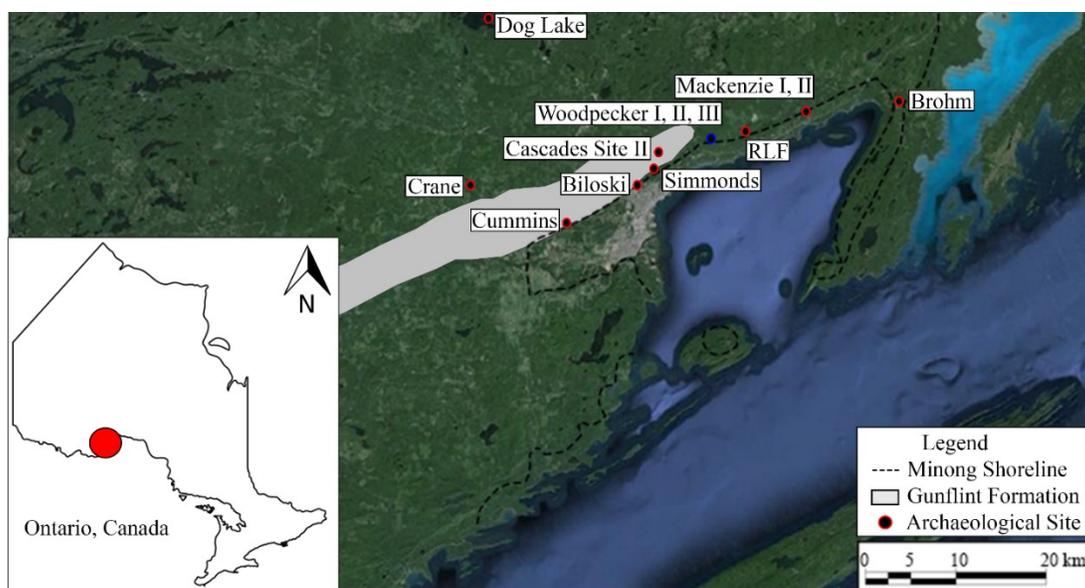
Limited site survey within Northwestern Ontario has resulted in a relative absence of Early Holocene site data within the region. This issue is further compounded by the slowly accumulating, frequently disturbed, and podzolic soils within the Boreal forest that often result in mixed or indistinguishable cultural sequences (Hinshelwood 2004; Norris 2012; Pilon and Dalla Bona 2004). As aceramic sites in Northwestern Ontario generally consist of small lithic scatterings, providing chronological estimates based on diagnostic typologies is difficult and sometimes impossible (Hinshelwood 2004). Salvage excavations conducted in advance of development have provided a rare opportunity to investigate the lifeways of some of the earliest populations to inhabit the region.

The primary lithic resource utilized in this region at the time of occupation was taconite, an iron-rich silicate mineral found within the Gunflint Formation. Taconite represented the majority of lithics recovered from WP11, with a small occurrence of locally available chert and exotic sandstone. Due to the lack of visible stratigraphic sequences and poor soil deposition, the duration and frequency of re-occupation of the site is indeterminate. It should be noted that the site had been used in past years as a dumping ground with a small gravel road running through the east-central portion, making soil disturbances highly probable and further compounding stratigraphic chronological data.

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S1: Shoreline of Glacial Lake Minong in relation to several Paleo-Indian archaeological sites in the Thunder Bay Region. Proximity to Gunflint Formation materials is shown in grey. Modified from Fox 1975 and Julig 1990.

APPENDIX A

METHODOLOGY

A.1 INTRODUCTION

The following pages present the sequential methodology employed throughout this project. The sequence of methods reflect the attempt to off-set any possible researcher bias, a traditionally cited limitation within use-wear analysis. Results from the use-wear portion of the project were finalized prior to the analysis of results from the multi-analytical residue analysis. Additionally, comparative data was created through an experimental study completed prior to the project.

The preliminary phase included macroscopic analysis, macro-scale photography, tentative identification of the working edge, and low-powered microscopic analysis to confirm working edges and to document amorphous *in situ* residues prior to residue extraction. The extraction process prepared the artifacts for further use-wear analysis by simultaneously cleaning the working edge. Both micro-flake and feature analyses were completed during the use-wear analysis using low- and high-powered incident light microscopy.

The multi-analytical residue analysis was completed following the interpretation of use-wear damages. Stages of this process include additional incident light microscopic analysis of *in situ* residues, transmitted light microscopic analysis of extracted residues, biochemical testing, characterization with absorbance spectroscopy, and analysis via gas chromatography coupled mass spectroscopy (GC-MS).

A.2 PRELIMINARY ANALYSIS

A.2.1 Macroscopic Analysis

Macroscopic analysis took place following the completed sample selection. Total artifact size (maximum length, width, thickness), edge angle, lithic material type, and tool type (formal, informal, expedient) were recorded in this phase. Tentative working edges were identified through observation with a 16x hand lens. Each artifact was then photographed using and Canon T2i DSLR camera with a macrolens attachment.

A.2.2 Photomicrography

Each artifact was viewed under 15-97x magnification using a Nikon SMZ800 incident light microscope with a VarianII camera attachment (1.5x) and a Canon EOS70D digital camera. The location of tentative amorphous residues or contaminants were recorded on line drawings of each artifact. Amorphous residues located along the proposed working edges or hafting areas, particularly those partially covered by adhering sediments, were given priority throughout the scan.

A.3.0 SOLVENT SELECTION

Solvent selection within residue analysis varies widely depending on both the targeted residues (starches, fatty acids, proteins, etc), and the characteristics of the item being analyzed (i.e. lithics, ceramics, groundstone). The scope of the current study was broad, allowing for the identification of carbohydrates, starches, fatty acids, proteins, and resins. A broad scope such as this requires a flexible extraction solution which allows the removal of each of these compounds. Given the age of the sample study, damage to any residues present through oxidation or degradation was highly probable. These modified organic compounds result in higher polarities within mixed residues, resulting in the need for an equally polar extraction method (Cook 2016; Crowther 2015). The use of a tri-mixture ensures solvents of variable polarities were used.

The tri-mixture solvent solution, consisting of equal parts double-distilled water, acetonitrile, and ethanol, was selected because of its ability to meet the above criteria. Acetonitrile and ethanol are effective at removing hydrophobic compounds such as lipids, even when mixed with water (Lin et al. 2007). The concurrent use of water in a solvent mixture increases the overall polarity, allowing for the removal of compounds damaged by oxidation over time. Acetonitrile was selected due to both its miscibility with water and its capacity to dissolve amino and fatty acids. Ethanol is likewise miscible with water, and is effective at dissolving resin acids. This tri-mixture, capable of breaking down a variety of organic residues, was ideal for non-specific feasibility determination (Cook 2016; Crowther 2015).

A.4.0 EXTRACTION PHASE

A.4.1 Edge cleaning and residue extraction

Total working edge removals were completed for the majority of the artifacts. When possible, tentative hafting areas were submerged at the same time. Shorter sonication times were selected than those common within the literature to prevent a full removal of residues, should additional analyses be completed within the future.

Targeted areas were placed in suitably sized acid-washed sterile glass vessels. Tri-mixture was added in 200 μ increments until the targeted area was covered, and was then sonicated for 45 minutes. Solvent and solution ratios can be seen in Table A.1. Earlier experimental tests had been completed to ensure that the gentle vibrations caused in this stage did not cause additional damage to the tool edges or surfaces that could be confused with use-related damage.

Artifacts were air-dried for a period of one hour following extraction. The total extraction amount was recorded and transferred via sterile pipettes into 2ml acid-washed sterile crimp-top vials. An average of 50 μ l of each extraction was placed into a separate vial to be used for biochemical testing. An additional 2 μ l were placed onto a sterile glass slide, desiccated at room temperature, and mounted with PermountTM under a glass cover slip for later transmitted light microscopic analysis.

Table A.1

Solvent and total extraction amounts

Sample	<i>First Extraction</i>		<i>Second Extraction</i>		<i>Spot Extraction</i>	
	Solvent	Total	Solvent	Total	Solvent	Total
UN1	3.6ml	1.6ml	-	-	-	-
UN2	2.4ml	2.2ml	-	-	-	-
UN3	3.0ml	1.4ml	-	-	-	-
UN4	2.4ml	2.1ml	-	-	-	-
UN6	2.4ml	1.6ml	-	-	-	-
UN8	2.7ml	1.9ml	-	-	-	-
UN9	2.4ml	2.1ml	-	-	-	-
UN10	3.0ml	1.1ml	-	-	-	-
UN11	3.0ml	1.1ml	-	-	-	-
UN13	3.0ml	.75ml	-	-	-	-
UN14	3.0ml	1.85ml	-	-	-	-
UN15	3.0ml	1.1ml	-	-	-	-
UN18	1.5ml	1.1ml	-	-	-	-
UN19	3.0ml	1.7ml	-	-	-	-
UN21	3.0ml	.4ml	3.0ml	1.5ml	.2ml	Failed
UN23	1.5ml	1.1ml	-	-	-	-
UN24	3.0ml	.5ml	3.0ml	1.2ml	.2ml	Failed
UN25	3.0ml	1.1ml	-	-	-	-
UN26	3.0ml	1.4ml	-	-	-	-
UN27	3.0ml	.25ml	-	-	-	-
UN29	3.0ml	.9ml	-	-	-	-
UN30	3.0ml	1.9ml	3.0ml	1.5ml	.2ml	Failed
UN33	3.0ml	.2ml	3.0ml	1.2ml	.3ml	Failed

A.4.2 Contamination Protocols

Procedures were undertaken to prevent possible cross-contamination between samples and the lab environment. All glass slides were first cleaned with a 70% ethanol/water mixture and air-dried prior to use. A single slide was left uncovered within the laboratory for 48 hours in order to create a comparison for airborne contaminants. Between uses, each vessel was rinsed first with double-distilled water, and then with a 70% ethanol/water mixture. The vessels were air-dried completely, and then rinsed again with 10% hydrochloric acid and air-dried for one hour.

A.5.0 USE-WEAR ANALYSIS

Tentative working edges were confirmed or refuted during this phase through observation of micro-flake scars (shape, termination, distribution, frequency) and features present (nibbling, polish, striations, rounding, smoothing, etc). Images were taken using both low- and high- powered microscopy, and damage locations were recorded on line drawings of each artifact. Post-depositional or naturally forming damages were identified and excluded at this stage.

A.5.1 Low-powered analysis, recording techniques, and two methods of analysis

An average of 40x-50x magnification was used throughout the stage of analysis. Tentative working edges were microphotographed in entirety, spaced approximately 4mm or less apart. Each location was photographed between five and 20 times to capture all visible depths, and then stacked with ZereneStacker© software to provide clear and detailed images.

Analyses of micro-flake characteristics as well as features were completed. Each scar shape and termination type were recorded, in addition to their averages sizes and distributions. When appropriate, non-working edges (the surface and tentatively hafted areas) were analyzed at a later time with higher powered microscopy; this will be discussed further below. At the onset of this project, tool edge micrographs were analyzed in entirety. This process proved to be extremely time consuming, and was subsequently shortened by analyzed representative images selected from central and outer areas of use.

Features include nibbling, polish, smoothing, rounding, striations, stepping, and crushing. Definitions of these features can be found in Chapter 2. Each of these could be further characterized by the degree of their presence, i.e. light to heavy, narrow to wide, etc. Features and micro-flakes were recorded for each of the selected images to ensure continuity of results.

A.5.2 High-powered Analysis

Features typically required addition high-powered analysis to be adequately analyzed. Increasingly detailed description of polish and striations specifically were only possible with higher magnification. Magnifications of 100x, 200x, and 500x were used with an Olympus BX51 incident light microscope. All artifacts with preliminary indications of hafting, i.e. wear on non-working edges or surfaces of the artifact, were always observed with higher magnifications. Images locations were recorded on line drawings of each artifact.

A.6.0 RESIDUE ANALYSIS

Each artifact in this project was subjected to both in-situ incident light and transmitted light microscopy, biochemical testing, absorbance spectroscopy, and gas chromatography coupled mass spectroscopy. The order shown here reflects the order in which these stages were completed.

A.6.1 Gas Chromatography coupled Mass Spectrometry

Immediately following the extraction phase, residues set aside for GC-MS testing were frozen at -83°C for a 24 hour period, ensuring that all liquid components of the extraction were solid. While in a solid state, they were placed into a LabConco Freeze Dryer for a three day period, or until desiccation was completed. Two extractions proved difficult at this stage for an unknown reason, and took over five days to fully evaporate.

Once desiccated, the samples were derivatized with 600µl of LCMS-grade acetonitrile and 100µl of BSTFA (bis(trimethylsilyl)trifluoroacetamide) (Sigma-Aldrich). Each vessel was evacuated using nitrogen sprays for thirty seconds and were immediately sealed with crimp-top caps. Samples were incubated at 120° C for 30 minutes on a Baxter Scientific Multi-Block. Though the derivitizing agents were effective for up to 24 hours, samples were typically submitted for GC-MS analysis within 30 minutes.

A Varian model 450 gas chromatograph was coupled with a Varian model 300-MS quadrupole mass spectrometer equipped with a Factor Four™ capillary column. Helium was used as the carrier gas and samples were introduced via splitless mode in an autosampler with the injection port at a temperature of 270° C. The column temperature was initially held at 50° C for 2 minutes before being increased to 155° C at a rate of 8° C per minute. Temperature was again increased to 275° C at a rate of 40° C per minute and held for nine minutes. The ion source was set at 200° C under electron ionization (EI) conditions, producing ionization energy of 70 eV. A scan range of 40 to 500 m/z was used, with a GC-MS interface temperature set at 266° C.

Output files were analyzed using Varian MS Workstation (Version 6) and the NIST98 Mass Spectral Database. Any peaks above background static were recorded. A minimal threshold was not in place due to the highly degraded environment of the study area in order to ensure the collection of even the smallest amount of data. Compounds of potential archaeological relevance were matched to compounds from the database whenever possible. When a suitable match could not be determined in this manner, the compounds were examined manually to ensure a positive identification. Chemicals that were determined to be contaminants were noted and excluded from the resulting archaeological interpretation.

A.6.2 Transmitted Light Microscopy

Slides created immediately following residue extraction were analyzed with an Olympus BX51 stereoscopic microscope, with magnifications of 100x, 200x, and 500x. Organic structures with identifiable characteristics were photographed and compared with images within the literature for identification. When matches could not be made, a small number of experimental reference slides were created for comparison (i.e. use-wear flakes, horn, bone, and antler). Modern or common environmental

structures were recorded as contaminants, while degraded or unusual structures (not commonly found within burial environments) were recorded as tentatively authentic residues.

A.6.3 Incident light microscopy for in situ residue analysis

Each artifact was scanned under high-powered incident light with an Olympus BX51 stereoscopic microscope. These scans included working edges, non-working edges, and tool surfaces. Priority was given to residues that were still visible on the cleaned edge of the tool, as well as those partially covered by adhering sediments. Amorphous residues were noted and recorded on line drawings of the artifacts. A detailed search of images available within the literature was completed to characterize *in situ* residues as closely as possible.

A.6.4 Biochemical Tests and Absorbance Spectroscopy

Biochemical tests were employed to determine the presence of carbohydrates, starches, fatty acids, and proteins. All four tests were used on each artifact within the sample set, and were optimized to require only 5 μ l of extraction sample. A secondary optimization allowed for immediate characterization with absorbance spectroscopy using 2 μ l of each sample.

The diphenylamide test was selected to determine the presence of carbohydrates, or simple sugars. The reagents were added to 5 μ l of sample solution and heated from ten minutes at 90°C. Positive results were indicated by a blue color change. The solution was then tested at 595nm. Characterization via absorbance spectroscopy was completed on each sample regardless of color change.

The presence of starch was determined with an iodine test. The sample was first heated at 60°C for 15 minutes. The reagent was then added, with a blue color change indicating a positive reaction. Solutions were then tested at 595nm.

The Solani and Sardoni test was used to determine the presence of fatty acids. The reagent was added to the sample solution and allowed to sit at room temperature for a period of 15 minutes. Positive results were indicated by a purple color change. The solution was then tested at 550nm.

Finally, the Bradford test was used to determine the presence of proteins within the sample. The Bradford reagent was added to the sample solution, vortexed for several sections to ensure adequate mixing, and allowed to sit at room temperature for 20 minutes. Positive results were indicated by a blue color change. The solution was then tested at both 595nm and 530nm.

Following the completion of all tests, it was found that the Bradford reagent reacts positively with acetonitrile, one of the three solvents used in the tri-mixture. As a result, all protein tests within this stage were positive, and unsuitable for inclusion within the interpretation. Time did not permit the tests to be re-done.

The degree of color change was then quantified via absorbance spectrophotometry. This process indicated the relative concentration of the targeted organic compound within the residue solution. A Bio-Tech Epoch Micro Plate Spectrometer was used to determine the amount of light absorbed at wavelengths specified by the test parameters. These parameters were determined within associated literature. Each test required 2µl of residue solution; two portions of each sample were submitted with each test.

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APPENDIX B

IN-SITU MICROGRAPHS:

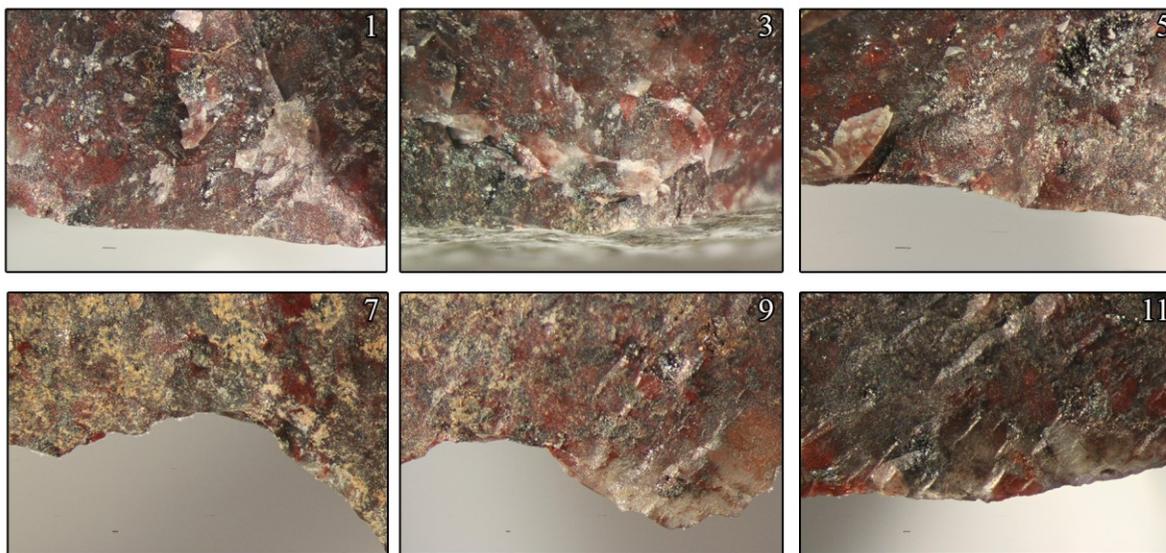
LOW AND HIGH-POWER IMAGES AND LOW-POWER DATA SHEETS

B.1 INTRODUCTION

The following pages contain all *in-situ* microscopic images included in the use-wear analysis. The section is formatted sequentially by sample number, beginning with UN1. Each individual ‘package’ of information consists of photographs of the dorsal and ventral surfaces of the tool, low- and high-powered microscopic images, and tables describing the wears observed in detail. Overview photographs of each artifact are located at the top of the page. Low-powered microscopic images are located immediately below this and labelled numerically. Matching numbers are located on the artifact image depicting the location of the damages. High-powered images, located below low-powered images, are labelled alphabetically, again with matching letters located on the overview image depicting location. Tables describing all wears present in the low-powered images concludes each package. Each description is matched with the numerical designations visible on the low-powered images. Table abbreviations are as follows: F (feather termination), H (hinge termination), S1 (step type 1 terminations), S2 (step type 2 terminations), and C (concave snaps).



Low-powered Micrographs



High-powered Micrographs

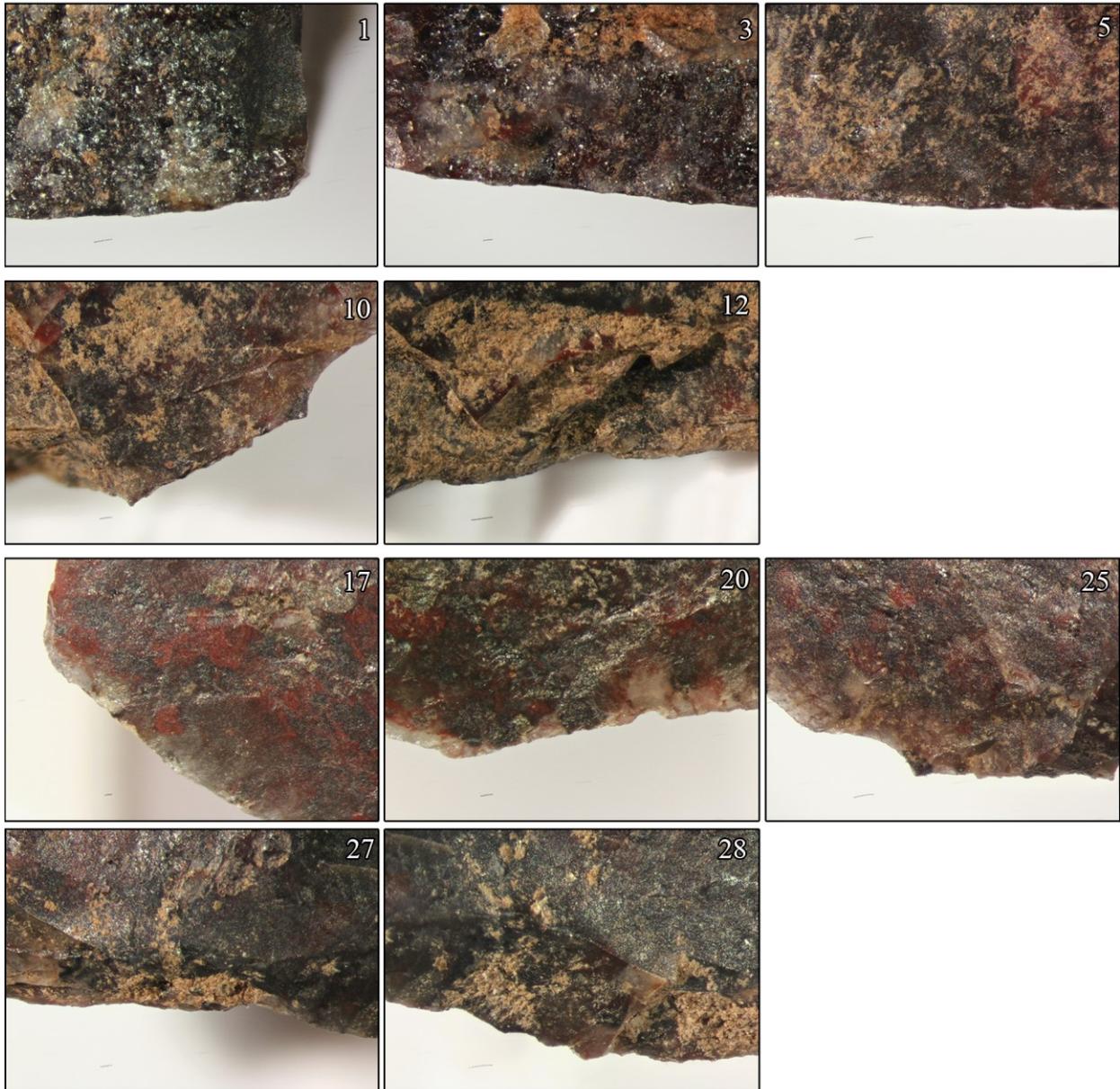


100x: Minor rounding and smoothing, with randomly distributed bright spots (a), thick and uneven polish along working edge, liquid-like in places (b), and bright polish associated with micro-topographic peaks within 1mm of the working edge (c).

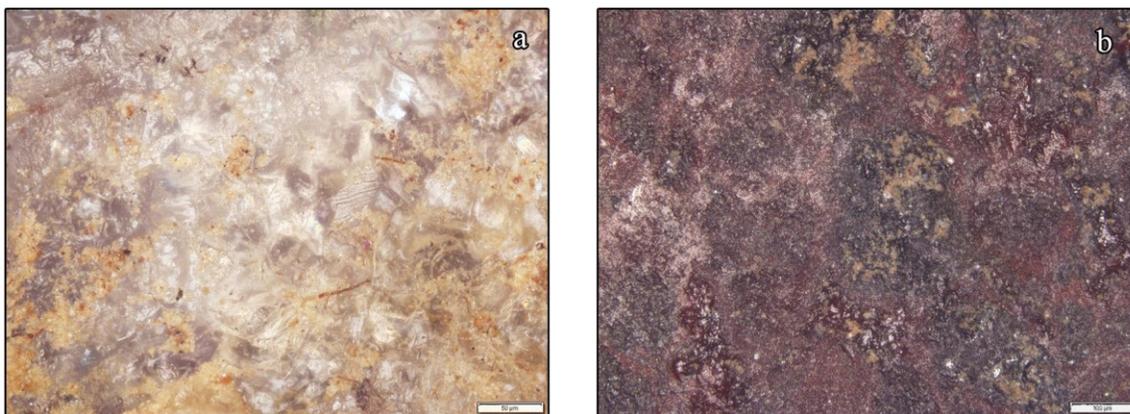
UN1 Micro-flake Analysis						Feature Analysis		
Ref		F	H	S1	S2	C	Feature	Description
1d	Scalar		3				Smoothing	Minor, even; larger S2 covers not fully removed
	Triangular		5				Polishing	Matte, evenly distributed
	Trapezoidal		4		1			
3d	Scalar		3				Smoothing	Minor to non-existent; no removal of S2 covers
	Triangular		17				Polish	Dull across surface, slightly bright but patchy along larger stepping and S2 covers
	Rectangular		2					
	Half-moon		3					
5d	Scalar		12				Smoothing	Minor and even; S2 covers not removed
	Triangular		19				Polish	Bright and patchy along outer two thirds of frame
	Trapezoidal		2				Stepping	Small region, single layered, along working edge
7v	Scalar		5	2			Smoothing	Minor to moderate, evenly distributed
	Triangular		7				Polish	Limited to highest micro-topography; very localized, patchy, bright spots
	Trapezoidal				1			
9v	Scalar		2		1		Nibbling	Mixture of broad and narrow distributed across frame
	Triangular		14				Smoothing	Minor to moderate; majority of S2 covers removed
	Trapezoidal		1		1		Polish	Limited to higher micro-topography, fairly localized; bright but patchy
							Stepping	One longer section, single layer, S2 fracture; cover not fully removed
11v	Scalar		4	3			Nibbling	Minor and very small across edge
	Triangular		7				Smoothing	Minor and evenly distributed
	Trapezoidal		2				Polish	Very dull and patchy across surface; bright but patchy along higher micro-topographies



Low-powered Microscopy



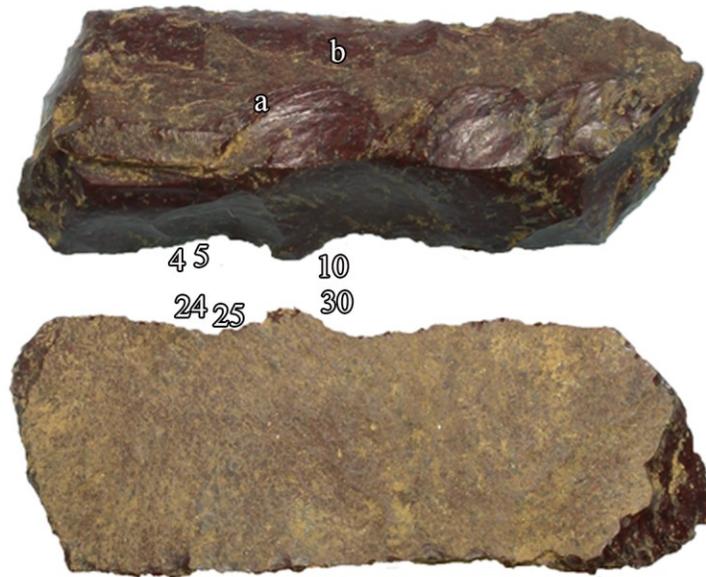
High-powered Microscopy



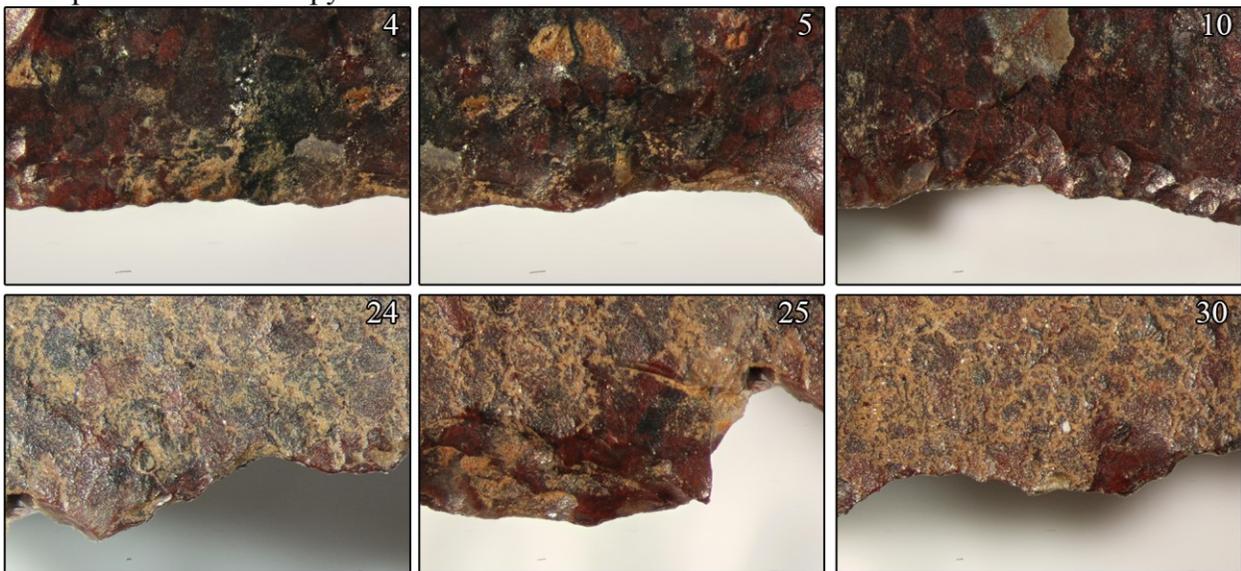
Mixture of amorphous and crystallized translucent white residue mixed with sediments, possible striations indicated by circle (a); clean, amorphous residues unevenly distributed across non-working edge (b).

UN2 Micro-flake Analysis						Feature Analysis		
Ref		F	H	S1	S2	C	Feature	Description
1d	Triangular			7			Smoothing Polish	none, no removal of S2 covers Very uneven due to topography, heavy on peaks
3d	Scalar		3				Rounding	Small amount, only near edge
	Triangular		5	3			Polish	Less uneven, limited to edge or further away from edge (central region clear)
	Trapezoidal		2	1			Striations	Tentative left oblique on level area, small and thin
5d	Scalar		6	5			Rounding	Uneven, very light along furthest edge
	Triangular		3				Polish	Light, uneven, patchy over surface, even around edge
	Trapezoidal			1				
10d	Scalar		3	1			Nibbling	Fine along protruding plateau, 250um
	Triangular		17	4			Smoothing	Complete removal of S2 covers
	Trapezoidal		4	2				
12d	Scalar			1			Smoothing	Minor delineation of grains
	Triangular			4			Stepping	Finer than 11d
	Trapezoidal	1						
	Half-moon					14		
17v	Scalar			7			Smoothing	Homogenous, no grain delineation
	Rectangular		5				Polish	Dull but even
	Trapezoidal		1					
	Half-moon				1			
20v	Scalar	1	1	4	2		Polish	Limited to peaks
	Triangular		5					
25v	Scalar		1	2			Polish	Even
	Triangular		8	1				
	Trapezoidal		1					
27v	Triangular		5				Polish	Even, similar to sand polish

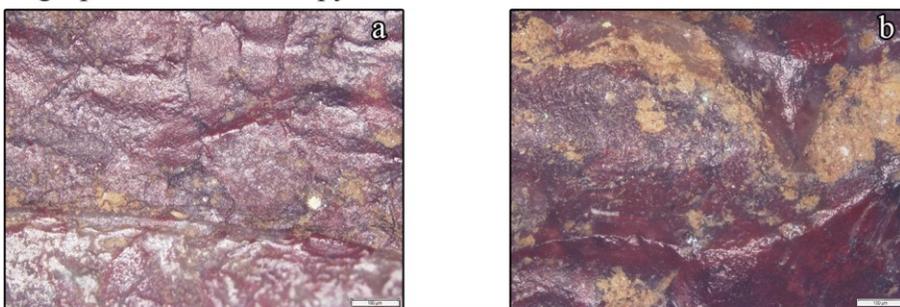
	Rectangular	3		Stepping	Minor, possibly from manufacture
	Trapezoidal		1 2		
28v				Smoothing	Very minor delineation of grains
	No significant scarring			Polish	Even, heavier on thicker areas, none on fresher scar faces



Low-powered Microscopy



High-powered Microscopy

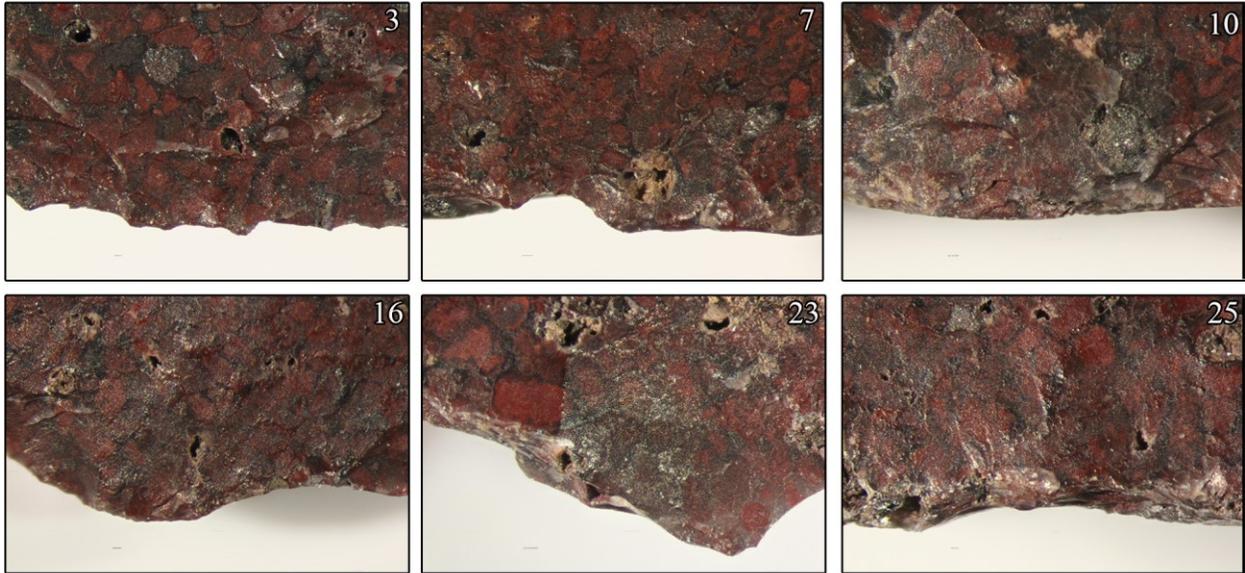


Contaminant fibers and differential smoothing between use-related and post-depositional wear (a); grinding along dorsal ridge with subsequent smoothing and polish limited to micro-topographical peaks (b).

UN3 Micro-flake Analysis						Feature Analysis	
Ref	F	H	S1	S2	C	Feature	Description
1d	Data unavailable					Smoothing	General, complete area
						Polish	moderate on peak, even when present
						Stepping	Light, fairly small area, possible PD or M
4d	Data unavailable					Nibbling	Fine, even, 250um
						Rounding	Minor on nibbled edge
						Smoothing	Very minor grain delineation, 2mm from edge
						Polish	Moderate on peaks, uneven
						Stepping	Minor, small area
5d	Data unavailable					Nibbling	Fine within broader crenellations
						Rounding	Minor around nibbled edge
						Polish	Moderate on peaks
						Stepping	Around curvature of protrusions, 3 layers
6d	Data unavailable					Nibbling	Fading, almost non-existent
						Rounding	Minor, limited to fading nibbled edge
						Polish	Even in and above manufacture scars, possible sand polish
						Stepping	Uneven and invasive, messy
7d	Data unavailable					Polish	Even excluding messy stepped area
						Stepping	Continuation of messy stepping
8d	Data unavailable					Polish	Sand polish
10d	Data unavailable					Nibbling	Moderate around bend
						Smoothing	No removal of S2 covers
						Polish	Only visible on large wear scars
						Striations	Tentative fine, slightly left oblique and perpendicular
11d	Data unavailable					Rounding	Minor, unevenly distributed, not in concave areas
ALL v	Data unavailable					Nibbling	Unequal throughout, mixture of fine to moderate
						Rounding	Minor along most of edge, less in concave sections of protrusions
						Smoothing	Grain delineation apparent
						Polish	Continuous along tool edge, fairly even. Likely sand polish.
						Stepping	None on ventral surface



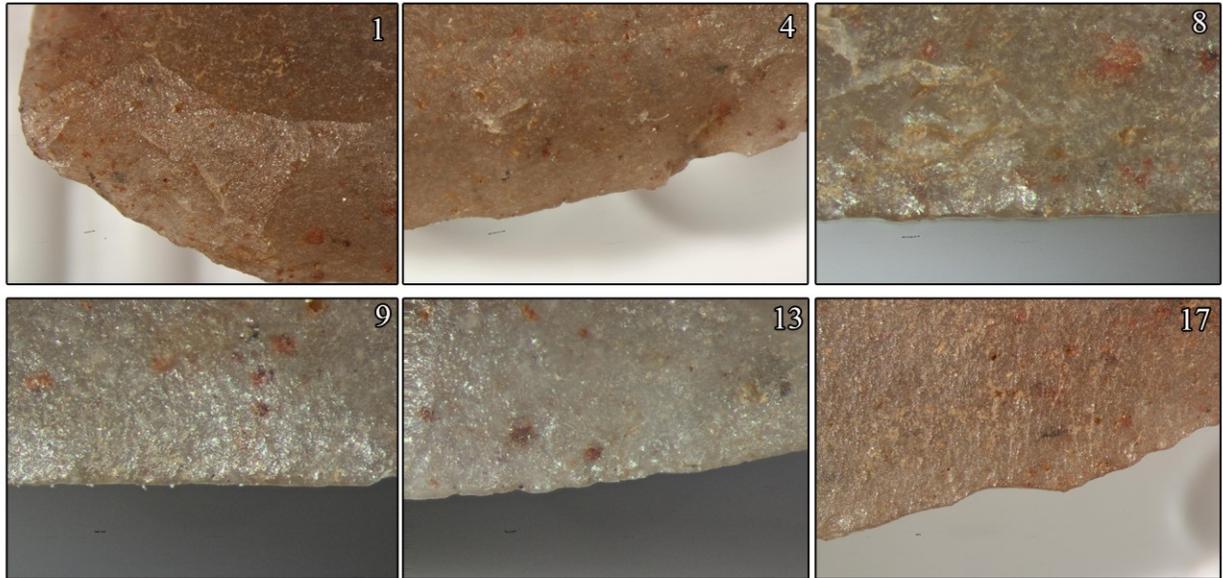
Low-powered Microscopy



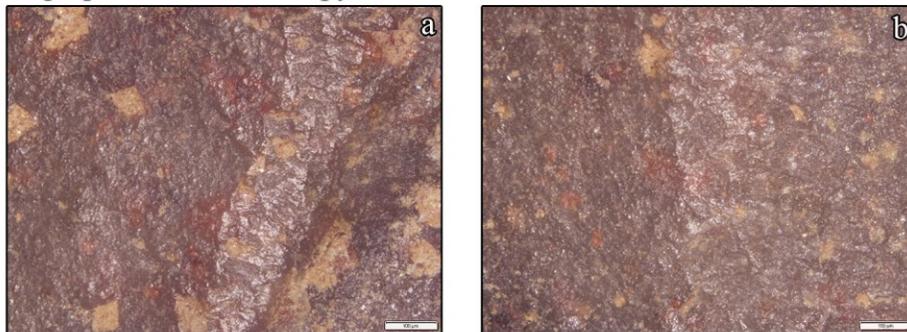
UN4 Micro-flake Analysis						Feature Analysis		
Ref		F	H	S1	S2	C	Feature	Description
3d	Scalar		1				Nibbling	Uneven mixture of fine to moderate with blocky corners
	Triangular		2	1			Rounding	Very minor on outermost edge
	Trapezoidal		3				Smoothing	Minor removal of S2 covers; grain delineation back from edge
							Polish	Heavy, even with 2mm of snapped edge
7d	Triangular		2				Rounding	Small amount of furthest edge, consistent amongst concavities
	Trapezoidal		4				Smoothing	Grain delineation starts to fade, general smoothing
							Polish	Uneven, concentrated on topographic peaks
							Stepping	Layers of 3-7, interior is rounded, nearer to edge is not
10d	Trapezoidal		3	1			Smoothing	Minor grain delineation without removal of S2 covers
							Stepping	Six incidences in area; innermost areas are rounded, outermost are not
16v	Triangular		3	4	2		Smoothing	Minor grain delineation
	Trapezoidal		4	4	3			
23v	Scalar			2			Stepping	Minor along thicker edges
	Triangular		3		2			
	Trapezoidal		3	1				
25v	Scalar		1		2		Crushing	Minor to moderate crushing of protrusions
	Triangular		4				Rounding	Minor along outermost edge
	Trapezoidal		2				Stepping	Minor along thicker edges



Low-powered Microscopy



High-powered Microscopy



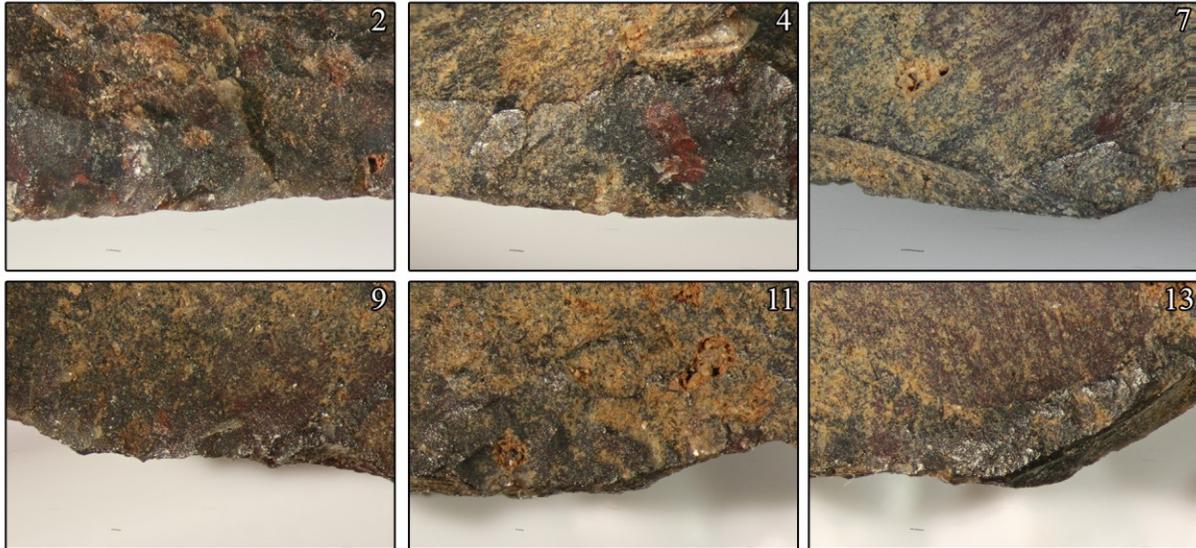
Differential polish and smoothing along shallow dorsal ridge (a); transition from dorsal ridge with damage to non-raised area (b).

UN6 Micro-flake Analysis						Feature Analysis	
Ref	F	H	S1	S2	C	Feature	Description
1d	No significant scarring					Nibbling	Minor, limited to left lateral edge of tool
						Rounding	Begins immediately after nibbling on lateral edge
						Smoothing	Homogenous topography
						Polishing	Minor on entire surface; liquid-like on lateral edge; strongest nearest working edge; greasy
4d	Scalar	3				Rounding	Moderate
	Triangular	2	2			Smoothing	Moderate on arrises, bumpy surface texture
	Trapezoidal	2				Polishing	Heavier nearest edge, lighter extending beyond micrograph borders. Bright and patchy.
8d	Triangular		1	1		Nibbling	Heavier than seen in 1d with more rounding, fine to medium spacing.
	Trapezoidal	2				Rounding	Minor
	Irregular		2			Smoothing	Minor
						Polishing	Bright and patchy, uneven; bright spots visible. Less invasive than seen in 4d.
9v	Scalar	4	2			Nibbling	Uneven, ranges from broad to fine. Fine is likely PD.
	Trapezoidal		2			Rounding	Minor
	Half-moon		3			Smoothing	Fine pitting over surface, fairly homogenous
						Polish	Uneven, very patchy and possibly pitted
13v	Scalar	6				Nibbling	Fairly even, right-crested
	Triangular	1				Rounding	Minor within nibbled areas
	Trapezoidal	2		2		Smoothing	Homogenous topography, but not smooth
	Half-moon	1				Polish	Bright spot near nibbling; uneven; slight color change between edge and surface
17v	No significant scarring.					Nibbling	Moderate to broad, uneven. Light rounded. Finer and increasingly jagged along right lateral edge of frame.
						Rounding	Very minor on outermost edge, less near scarring
						Smoothing	Heavier near lateral edge
						Polish	Heavier toward lateral edge; patchy; more pronounced on peaks and arrises

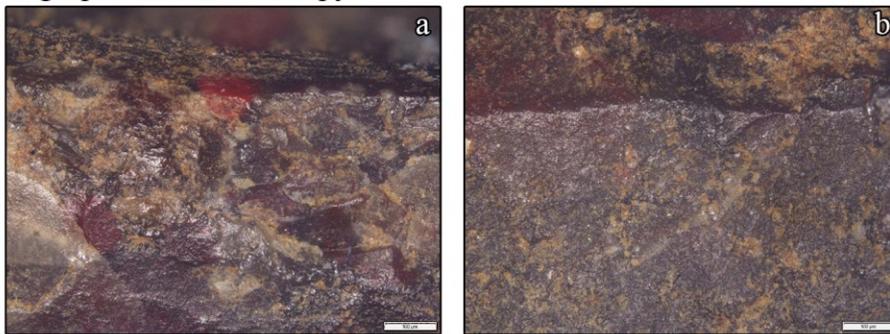
Note: 15d was not analyzed as a part of this study; however, it was noted that slightly right oblique striations were visible on this portion of the tool alone.



Low-powered Microscopy



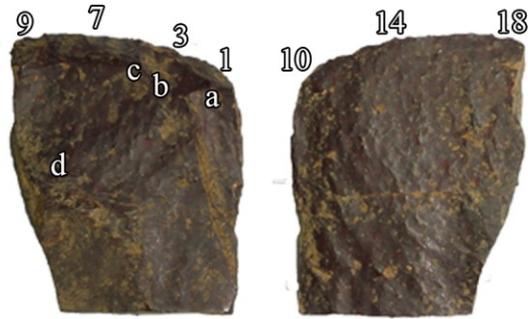
High-powered Microscopy



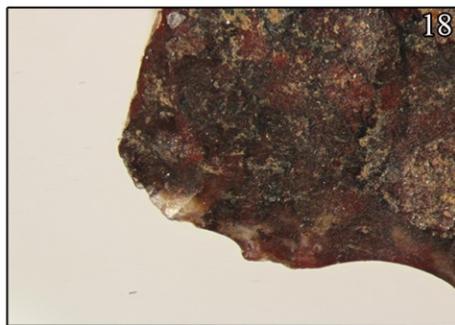
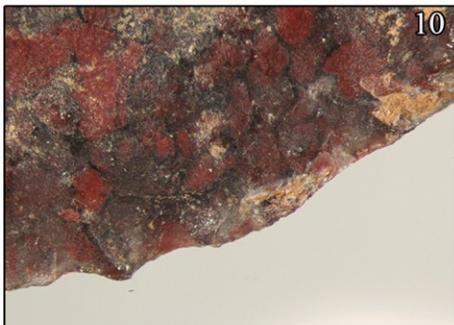
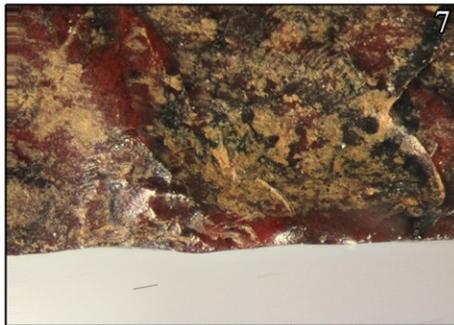
Ground edge with minor smoothing, light polish on ridges (a); un-ground edge for comparison (b).

UN8 Micro-flake Analysis						Feature Analysis		
Ref		F	H	S1	S2	C	Feature	Description
2d	Scalar		2				Nibbling	Fine, fairly even, dull
	Triangular		4	9			Rounding	Minimal, limited to nibbled regions
	Trapezoidal		5	7			Smoothing	Minor-moderate, heavier near working edge. Grain levelling present; incomplete removal of S2 covers
							Polishing	Bright spot, resinous but dull
							Striations	6+, very fine, parallel to working edge
4d	Scalar		1				Rounding	Very minor
	Triangular			4			Smoothing	Grain levelling present, no removal of S2 covers
	Trapezoidal		2	3	1	1	Polish	Bright, heavy on working edge; dull and uneven everywhere else.
	Irregular		1				Striations	Left oblique, very fine, clustered, small amount right oblique. Tentative and parallel to working edge away from edge.
	Half-moon		2					
7d	Scalar		3				Smoothing	Moderate grain levelling
	Triangular		5	3			Polish	Bright and heavy on edges of manufacturing scars, extends 200um below ridge on working edge.
	Trapezoidal		10				Striations	Tentative left oblique, fine, extensive. Possible working edge parallel further up edge.
9v	Scalar		5	1				
	Triangular		5	1				
	Trapezoidal		1	5	1			
			(xl),		(xl)			
			9					
	Half-moon		1					

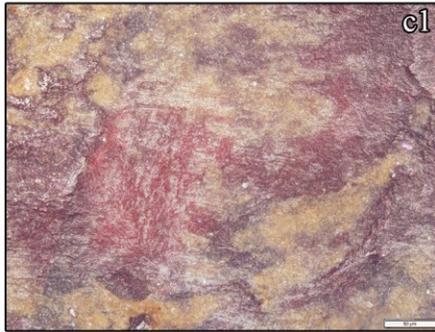
Note: 11v and 13v did not show significant micro-flaking damages.



Low-powered Microscopy



High-powered Microscopy



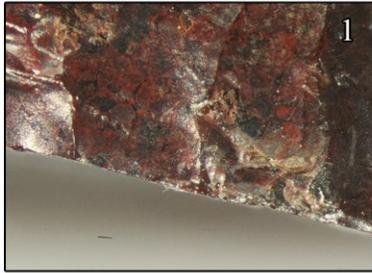
Glossy, liquid-like polish with minor pitting, 100x (a); cluster of left oblique striations superimposed on residue, 100x (b); additional cluster of striations, 100x (c1); closer view of c1, 500x (c2); heavy rounding and bright polish along the dorsal ridge (d).

UN9 Micro-flake Analysis						Feature Analysis		
Ref		F	H	S1	S2	C	Feature	Description
1d	Scalar		5				Smoothing	Grain levelling of valleys, incomplete removal of S2 covers
	Triangular		1				Polish	Bright and moderate over thicker edge; bright spots within scalar hinge scars on edge; liquid-like to the right of this.
	Trapezoidal		1		1		Striations	Tentative parallel or sharp right oblique near left lateral edge of tool
	Half-moon		1					
3d	Scalar			1			Crushing	Minor, 2mm area.
	Triangular			5			Smoothing	Minor, covers entire area excluding area above crushing
	Rectangular			1			Polish	Light, limited to ridges, uneven on working edge
	Trapezoidal			5			Striations	Tentative right oblique, sharply angled; right of crushed area
							Stepping	Minor, 2-3 layers in semi-crushed area.
7d	Scalar		2				Smoothing	Moderate grain elevation excluding 1-2 mm of the edge
	Triangular		10		1		Polish	Light, bright, more concentrated on ridges; three bright spots in thinner sections closest to edge.
	Rectangular		1				Stepping	Minor, 2-3 layers in 1-2mm section
	Trapezoidal		11					
9d	Scalar		2				Smoothing	Heavy across surface
	Triangular		3				Polish	Dull and patchy on left portion of micrograph; becomes increasingly liquid-like to the right
	Irregular		1				Striations	Tentative right oblique on fractured lateral edge
							Snap fracture	Left lateral tool edge; continued use after original breakage (scarring on edge and heavy polish).
10v	Scalar		1				Nibbling	Moderate to broad, even; finer is within broader sections; none is jagged
	Triangular		4				Crushing	Minor in one small, 1mm section.
	Trapezoidal		6				Smoothing	Minor grain delineation, no visible S2 fractures
							Polish	Light and dull; bright and patchy on thicker portions of working edge
14v	Scalar		4				Nibbling	Moderate to broad, not jagged. Right oblique cresting
	Triangular		2				Smoothing	Minor
							Polish	Dull and patchy everywhere; heavier on thicker portions of edge. Single uneven bright spot near edge.
							Striations	Sharply angled right oblique prior to polish
18v	Scalar			2			Nibbling	Very uneven, varied in size; edges slightly rounded. Possible PD intermixed.
	Triangular		1				Rounding	Limited to nibbled edges

Trapezoidal	1	Smoothing	Homogenous topography, but not smooth
Half-moon	2	Polish	Dull on ridges near broken edge
		Striations	Tentative very fine striations parallel to the edge; left and right obliquely angled present as well.



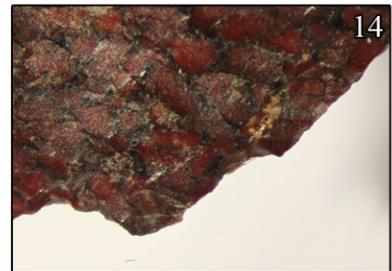
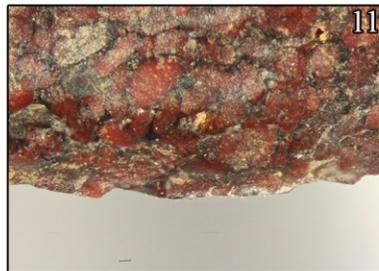
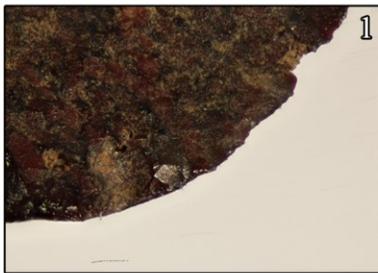
Low-powered Microscopy



UN10 Micro-flake Analysis						Feature Analysis		
Ref		F	H	S1	S2	C	Feature	Description
1d	Scalar		1		2		Crushing	Minor
	Triangular		6				Smoothing	Even excluding crushed area
	Trapezoidal		4	4	5		Polish	Moderate, bright on central ridge; two large bright spots; very bright smaller spot within depression
							Stepping	Small 'crushed' area, difficult to distinguish layers, 2-4 layers.
5d	Triangular		6	1			Rounding	Very minor, outermost edge
	Trapezoidal		6	4			Smoothing	Moderate to heavy on raised surfaces, minor on lower portions.
							Polish	Heavy along lower depression, liquid-like, uneven; moderate along remainder, bright spot on thickest portion of edge
							Stepping	Minor, 2-3 layers, slightly smoothed and rounded
8d	Scalar		2				Rounding	Minor, limited to small protrusion
	Triangular		5				Smoothing	Even excluding large scar areas
	Trapezoidal		2		2		Polish	Bright, even on higher portions of edge; moderate to heavy and smooth on lower portions (liquid-like); uneven bright sheen closest to edge and along ridges
9v	Trapezoidal	1			1		Smoothing	Minor, not on ridges
							Polish	Minor, limited to a single ridge; dull sheen on outer lateral edge. Single small bright spot on working edge.
12v	Scalar		3				Smoothing	Heavier near lower portion, uneven
	Triangular		3				Polish	Bright, patchy over higher surfaces; brightest in midsection
	Trapezoidal		2					
	Half-moon		1	1		1		
15v	Scalar	1	1	3			Nibbling	Rough, jagged, moderate; evenly distributed
	Triangular		1				Smoothing	Uneven, heavier on lower portions of topography
	Trapezoidal	1					Polish	Bright, patchy on raised area; even but dull sheen on lower portion; liquid-like on outermost working edge
	Half-moon	1						



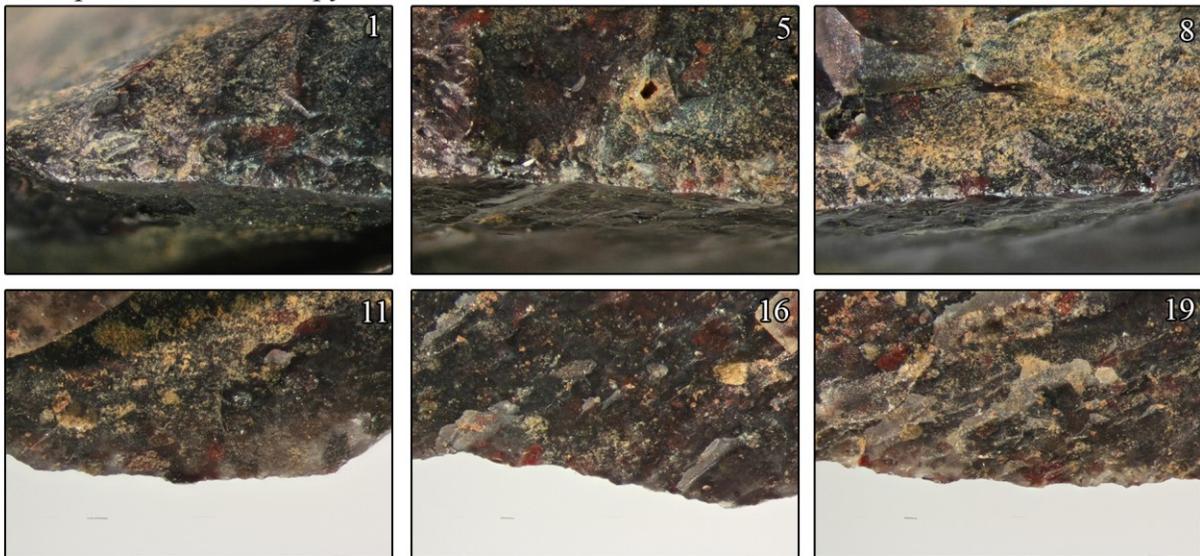
Low-powered Microscopy



UN11 Micro-flake Analysis						Feature Analysis		
Ref		F	H	S1	S2	C	Feature	Description
1d	Scalar	2	2				Rounding	Minor, outermost edge
	Triangular		2				Smoothing	Very minor, 2mm of edge
							Polish	Very dull over surface; bright on ridges and scar surfaces near edge
4d	Scalar	2		1			Nibbling	Broad, very shallow
	Rectangular	1					Smoothing	Extremely minor and uneven
							Polish	Minor on ridges further back from edge
7d	No significant scarring						Nibbling	Jagged edges, uneven
							Smoothing	Minor and uneven grain delineation of thin edges, minor smoothing on arrises
							Polish	Two ridges on thin corner; bright spot and heavy polish preceding corner; liquid-like beside
							Stepping	Minor on lateral corner
8v	Triangular		3				Nibbling	Fine, shallow, more frequent toward lateral edge
	Trapezoidal		4				Smoothing	Differential: oolites more so than interstitial silica, i.e. raised interstitial grain boundaries
							Polish	Concentrated on raised interstitial boundaries, becoming more even toward frame limits; liquid-like closest the edge curved edge
11v	Triangular			1			Nibbling	Uneven, moderate; single left crested, not jagged
	Trapezoidal		1				Smoothing	Differential interstitial grain boundary smoothing
	Irregular			1			Polish	Dull sheen across surface excluding apex of curve; brighter on outermost edge; heavier on grain faces than interstitial areas
	Half-moon		1	2				
14v	No significant scarring						Nibbling	Jagged, small, evenly spaced
							Smoothing	Moderate, even excluding lower worn down grains; differentiation between grains and interstitial boundaries
							Polish	Uneven, right oblique bright spots; heavier uneven near edge, most across surface



Low-powered Microscopy

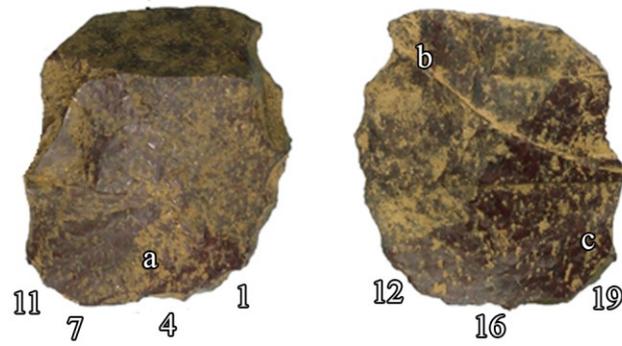


High-powered Microscopy

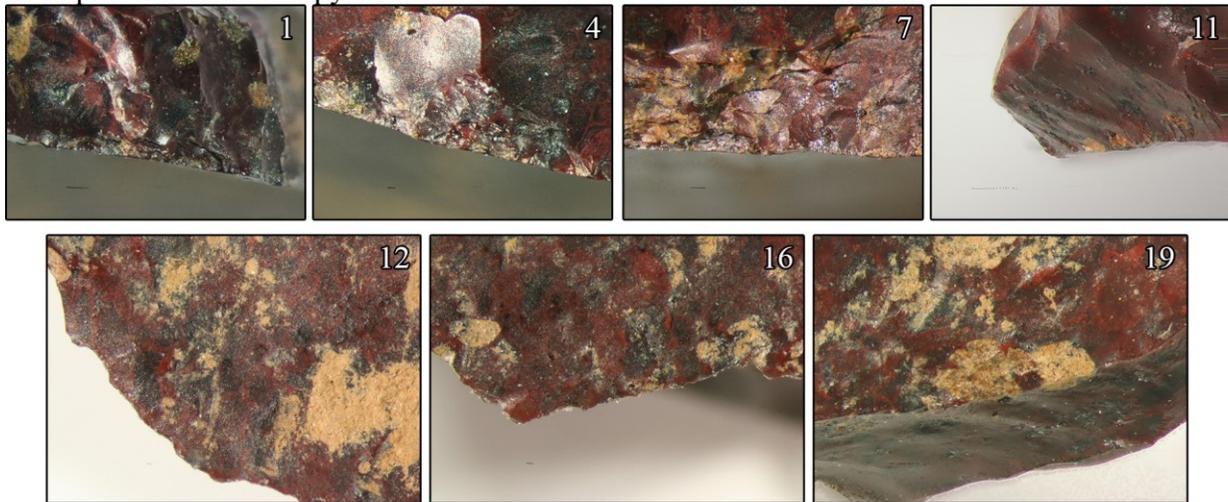


Heavy, patchy polish, 500x (a); brightly, patchy polish with moderate to heavy smoothing, 100x (b); and patchy polish mixed with orange, amorphous residue, 100x, (c).

UN13 Micro-flake Analysis						Feature Analysis		
Ref		F	H	S1	S2	C	Feature	Description
1d	Scalar		6				Smoothing	Very minor
	Triangular		8				Polish	Very minor
	Trapezoidal		11				Stepping	very minor stepping in 2-3 areas, partial removals of S2 covers
5d	Scalar		5		2		Smoothing	Minimal to moderate
	Triangular		10	1			Polish	Dull sheen on ridges; brighter on single area of working edge
	Trapezoidal		11		1		Stepping	Three layers
8d	Scalar		8				Smoothing	Mild, insufficient cleaning to determine extent
	Triangular		3		8		Polish	Dull, patchy sheen on surface and ridges
	Trapezoidal				15			
11v	Scalar	1	1				Nibbling	Fine, even
	Triangular		2				Smoothing	Heaviest nearest edge, grain levelling
	Trapezoidal		2				Polish	Matte, minor
16v	Triangular		1				Striations	Several, right oblique, fine
	Trapezoidal		1				Nibbling	Uneven, fine to moderate, jagged
	Half-moon			2			Smoothing	Even surface, incomplete removal of S2 covers
19v	Scalar		4				Polish	Uneven matte polish, minor
	Triangular		5				Nibbling	Uneven, fine to moderate, jagged
	Trapezoidal		6				Smoothing	Minor, very uneven, no removal of S2 covers
							Striations	Tentative right oblique striations



Low-powered Microscopy

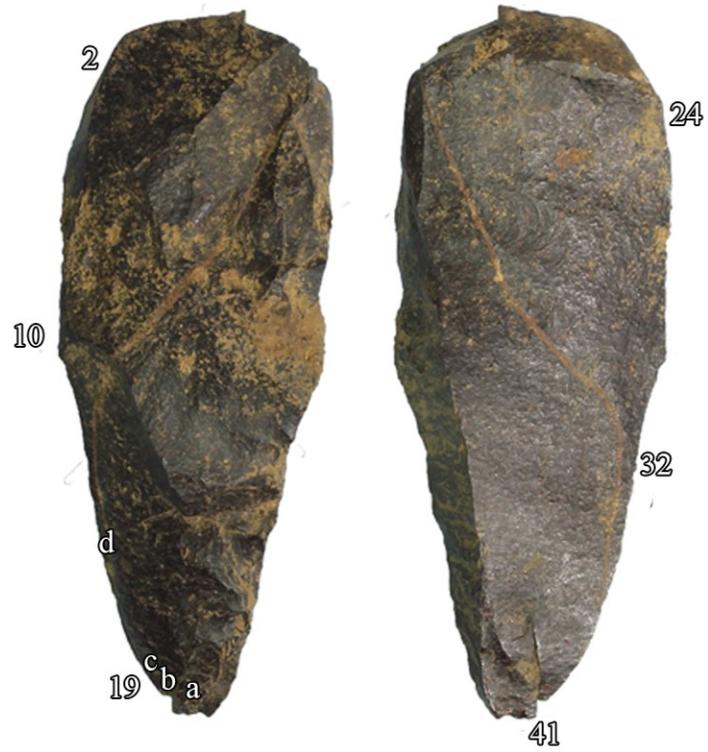


High-powered Microscopy

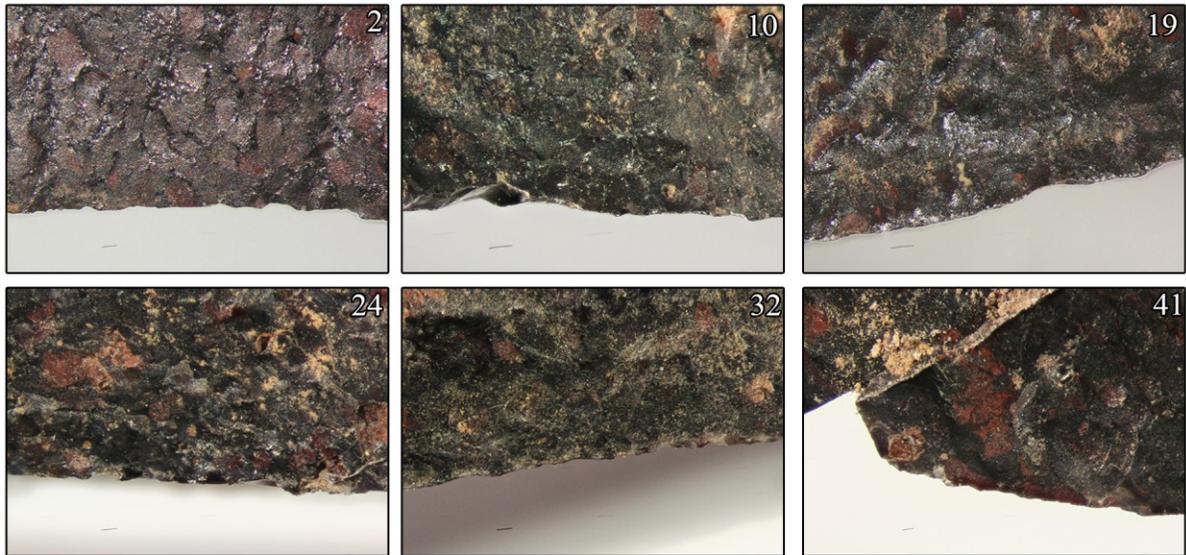


A large number of striations along the dorsal face of the working edge, 100x (a); glossy polish overlying an area with heavy smoothing, 200x (b); bright and continuous polish limited to the outer ventral edge, indicative of hafting, 100x (c).

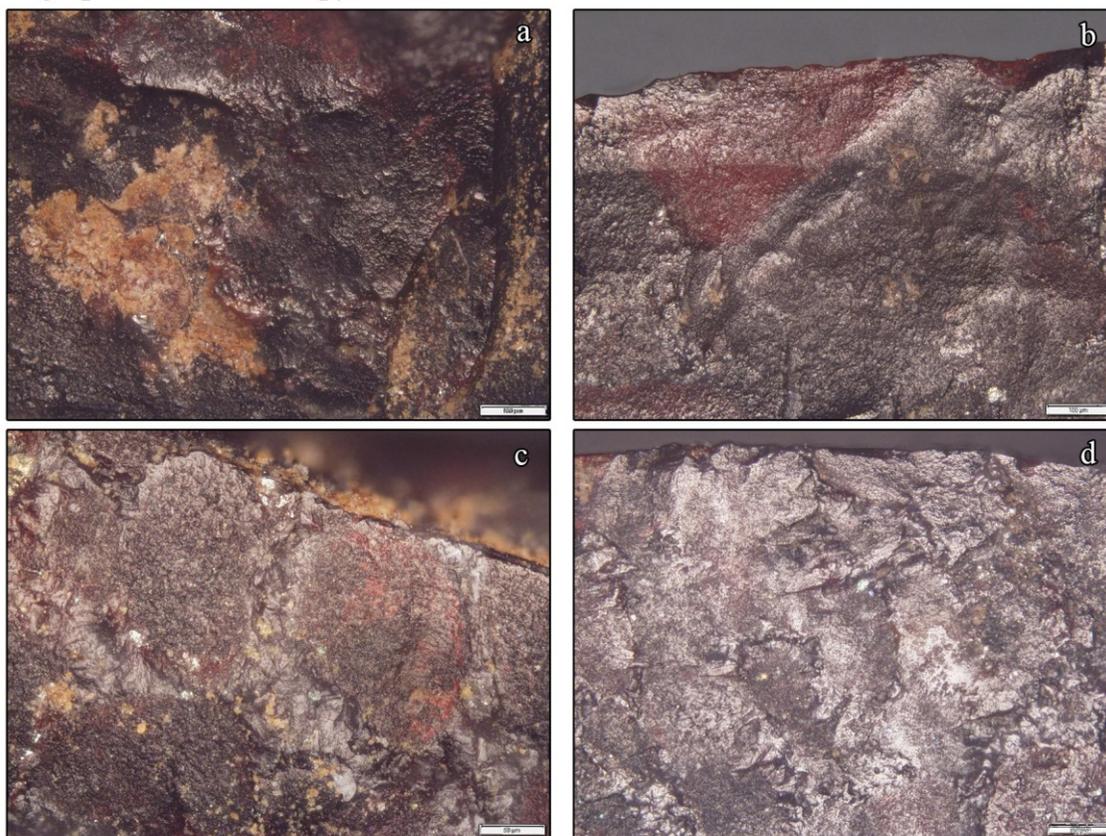
UN14 Micro-flake Analysis						Feature Analysis		
Ref		F	H	S1	S2	C	Feature	Description
1d	Scalar			2			Rounding	Very minor, uneven
	Triangular		9				Smoothing	Uneven, but heavy; less visible on damaged area
	Trapezoidal		4	2			Polish	Even; bright spot on upper ridge, smaller bright spot between stepped areas; less bright and moderate sheen above damaged edge, below heavier liquid-like polish
	Half-moon			2				
4d	Scalar		2				Smoothing	Moderate on brightly polished area; grain delineation present; stepped area slightly smoothed, S2 covers removed
	Triangular		13				Polish	Even, bright polish on interior plateau; duller sheen in area to the right; uneven bright polish covering stepped area and scar margins at times extending into vallies.
	Trapezoidal		22	1				
	Irregular		1					
	Half-moon		4					
7d	Scalar		7				Smoothing	Moderate to heavy, increases further away from edge
	Triangular		16				Polish	Even, dull over surface; brighter near edge and on interior plateaus; bright in a single vertical section of edge
	Trapezoidal		6	3				
11d	No significant scarring						Smoothing	Moderate until central margin, very heavy after
							Polish	Mix of bright and matte polish on moderately smoothed surface; dull but very heavy liquid-like polish on left side of central margin; brighter below horizontal margin closer to edge
12v	Scalar		5		1		Nibbling	Uneven, moderately sized. Possible PD
	Triangular		1				Smoothing	Minor to moderate; grain levelling without delineation
	Trapezoidal		1	1			Polish	Dull polish over surface excluding one small area parallel to edge
16v	No significant scarring						Nibbling	Fine to moderate, uneven, jagged
							Smoothing	Minor, grain levelling without delineation; heaviest within 1mm of edge
							Polish	Even, dull matte polish; 3 bright spots within 1mm of edge
							Striations	Very fine, perpendicular to edge
19v	No significant scarring						Smoothing	Minor, grain levelling without delineation over most of surface
							Polish	Right oblique oriented bright spots in six or seven areas
							Striations	Very fine, perpendicular to edge



Low-powered Microscopy



High-powered Microscopy

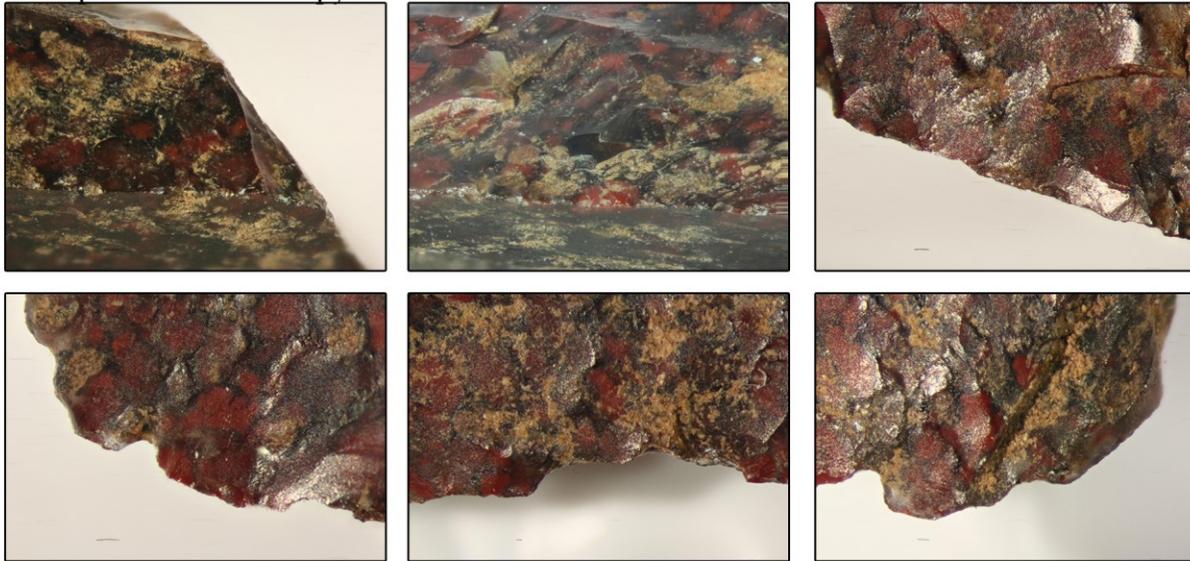


Minor polish on elevated microtopography and possible residue mixed with orange-brown sediment, 100x (a); clearly delineated polish on the dorsal working edge, 100x (b); an example of differential grain polishing or smoothing in which the interstitial silica matrix has been polished and the lower lying iron-rich silicates have been smoothed, but not polished to a similar extent, 200x (c); patchy moderate bright polish further removed from the working edge, 100x (d).

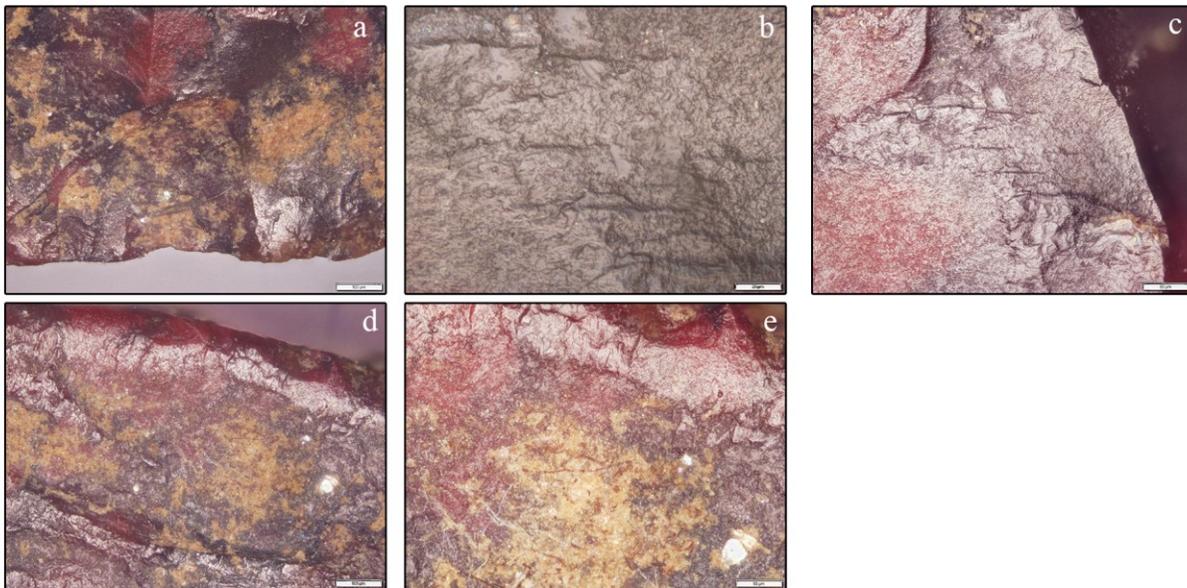
UN15 Micro-flake Analysis						Feature Analysis	
Ref	F	H	S1	S2	C	Feature	Description
2d	No significant scarring					Nibbling	Fine to moderate, uneven, slight rounded edges on some
						Smoothing	Moderate to heavy; grain delineation on most of surface, excluding 1mm of working edge
						Polish	Dull, even over whole surface; differential polishing on interstitial areas, bright; lessens closer to edge
						Striations	Very fine, tentative; mixture of parallel and right and left oblique; very faint
10d	Scalar	4				Nibbling	Fine to moderate, uneven, jagged
	Triangular	7				Rounding	Very minor on nibbled areas
	Trapezoidal	2				Smoothing	Moderate, uneven; minor grain delineation
	Half-moon	2		2		Polish	Differential over surface in a parallel banded formation, heavier on higher peaks; unevenly distributed bright spots; dull sheen further away from working edge
						Snap	Large, singular
19d	Scalar	6	2			Rounding	Moderate
	Triangular	1	6	5		Smoothing	Uneven over surface
	Trapezoidal	4	4	8		Polish	Differential; fairly even matte over surface, brighter and moderate on all ridges; brighter near working surface
	Half-moon	3				Striations	Tentative left oblique, very fine striations 2-3mm away from edge
24v	No significant scarring					Nibbling	Uneven, jagged, fine to moderate
32v	No significant scarring					Nibbling	Fine to moderate, uneven but continuous
						Rounding	Moderate within .5mm of edge
						Smoothing	Heavy to moderate
						Polish	Even, matte; heaviest near edge
						Striations	Tentative left and right oblique, unevenly distributed; both near edge and 2-3mm back
41v	Triangular	1	1		2	Nibbling	Fine, shallow, evenly spaced
	Trapezoidal	3	1	1		Rounding	Moderate, heavy within .5mm of edge
						Smoothing	Moderate, grain levelling without delineation excluding very minor presence away from working edge
						Polish	Very dull limited to a single small area
						Snap	Along natural fault on lateral corner, polish but not rounded



Low-powered Microscopy

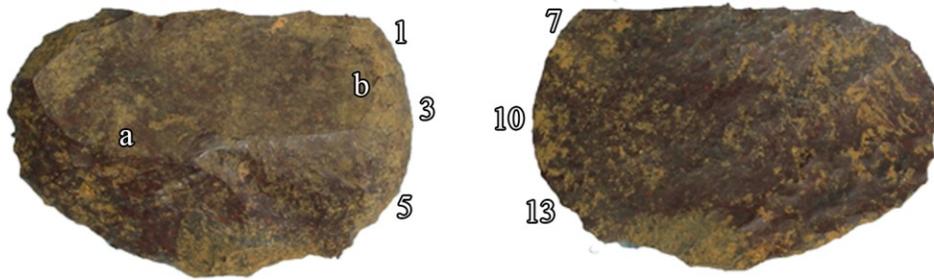


High-powered Microscopy

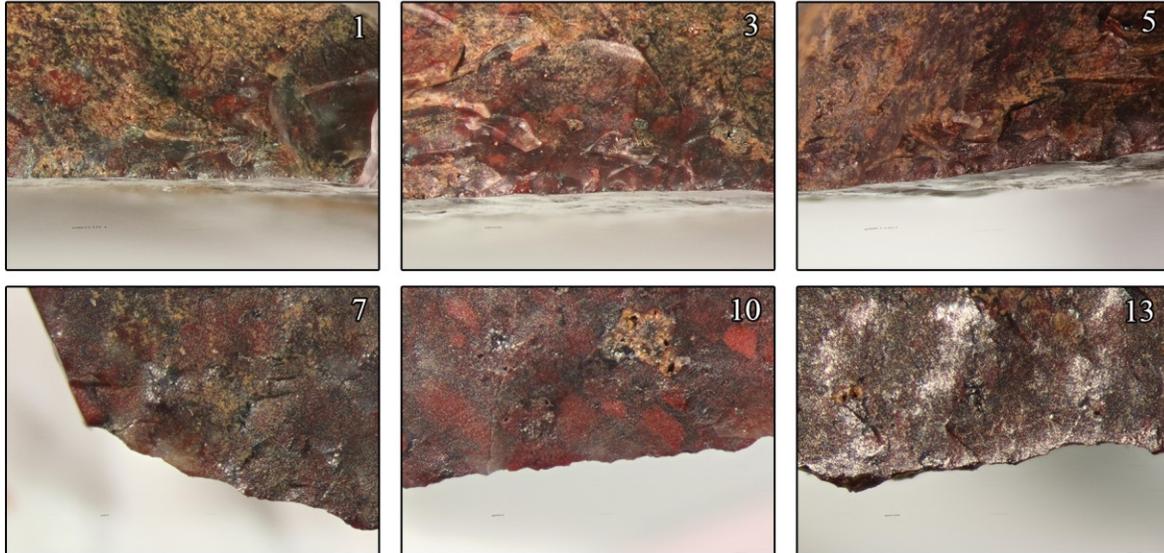


Microflaking along the working edge with polish on micro-topographical peaks, unevenly distributed, 100x (a); evenly distributed bright polish with several horizontal striations, 100x (b); previous image at 500x magnification (c); rounding, polish, and micro-striations indicative of hafting on the left lateral ventral surface at 100x (d) and 500x (e).

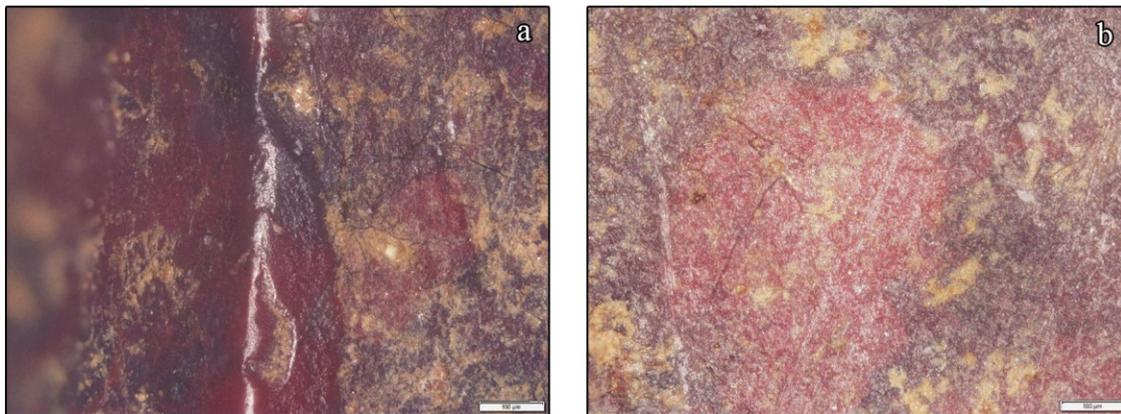
UN18 Micro-flake Analysis						Feature Analysis		
Ref		F	H	S1	S2	C	Feature	Description
1d	Scalar			2			Smoothing	Moderate over surface, heavy on working edge
	Triangular			3	2		Striations	Numerous in horizontal cluster 1mm away from edge, very slightly left oblique
	Trapezoidal			7	5			
3d	Scalar		2				Nibbling	Minor, all hinged; fine and even
	Triangular		4	3	2		Smoothing	Moderate; minor grain levelling
	Trapezoidal		10	1	3		Polish	Heavy, even, dull; liquid-like and translucent, fades near the surface and becomes patchier
	Irregular		1				Stepping	Spaced evenly horizontally across edge, possible PD
6d	Scalar	3	4				Nibbling	Minor, uneven, fine
	Triangular		2				Smoothing	Very minor
							Polish	Heavy but uneven; two bright, even spots (above step fracture, on thicker edge); single large bright area but patchy over grains; remainder is matte sheen
							Stepping	Tentative faint, only visible in polished areas
8d	Scalar	3		2			Nibbling	Minor, uneven
	Triangular			1			Rounding	Limited to broken surface around larger concavity
	Rectangular		2				Smoothing	Minor to moderate, reduction of prominent arrises
	Trapezoidal	1		1			Striations	Many, horizontal; majority between 1-1.5mm of edge in polished areas; parallel or slightly left oblique angled to the edge
	Half-moon			2				
13v	Scalar		5	1			Nibbling	Uneven, mostly shallow
	Triangular		1				Rounding	Minor
	Trapezoidal		5				Smoothing	Heavier on outer edge, even grain topography
	Half-moon					1	Polish	Bright, patchy, trails on ridges; dull matte sheen over majority of surface; fades closer to edge
15v	Scalar	2					Nibbling	Very uneven
	Trapezoidal	1	1				Rounding	Minor around nibbled edges
							Smoothing	Heavy over homogenous area; decreases on prominent ridges
							Polish	Moderate to heavy, bright but patchy and uneven spot on raised ridge; bright sheen across remainder, fades in scars



Low-powered Microscopy

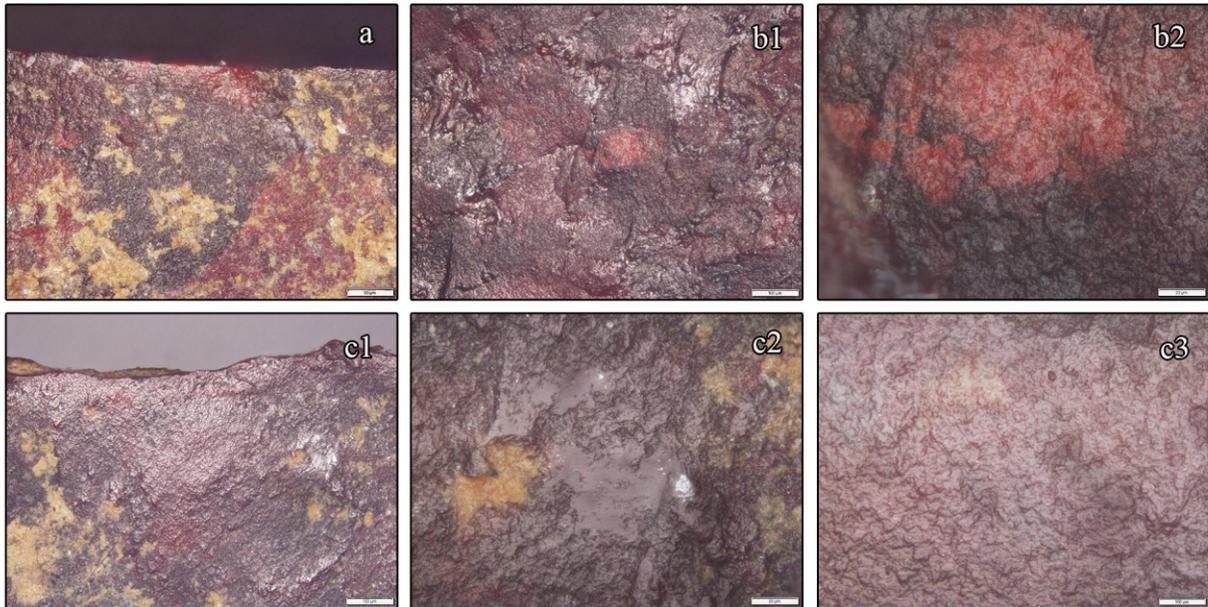
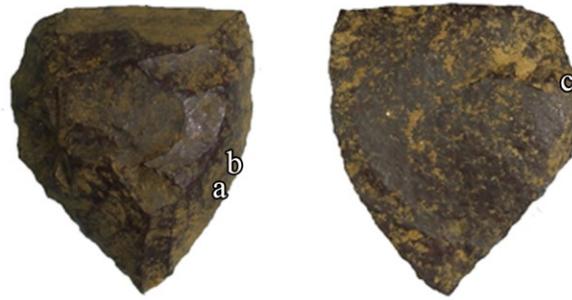


High-powered Microscopy



Rounded dorsal ridge with hinge scars, moderate polish, and heavy smoothing, 100x (a); striations oriented at right oblique angles and perpendicular to the working edge, deposited within the polish, 100x (b).

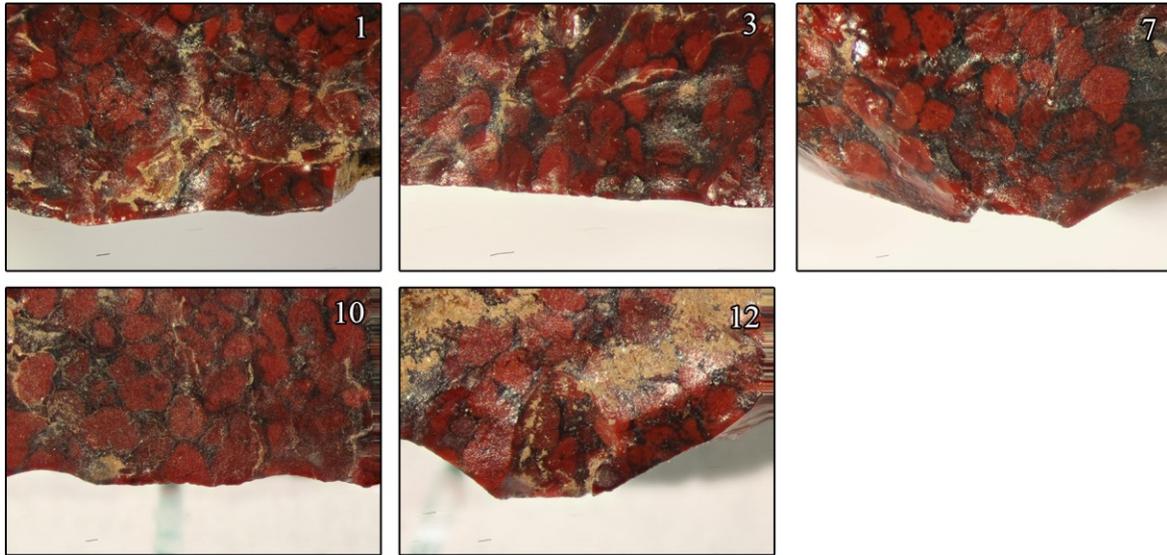
UN19 Micro-flake Analysis						Feature Analysis		
Ref		F	H	S1	S2	C	Feature	Description
1d	Scalar		3				Smoothing	Minor, heavier on lateral edge
	Triangular		4				Polish	Minor, even, brighter on ridges and margins
	Trapezoidal		8					
3d	Scalar	2	13	2	3		Smoothing	Moderate, heavier nearest edge; incomplete removal of S2 covers
	Triangular		9	1			Polish	Dull and patchy along margins; becomes increasingly smooth nearest edge
	Trapezoidal		4	1	2			
	Irregular		3					
5d	Scalar	3	5	2			Smoothing	Heavy and even in central region, moderate over rest
	Triangular		4				Polish	Heavy and liquid-like over central region; moderate and matte throughout area to the right, much lighter to the left; brighter within scar interiors along the edge
	Trapezoidal Half-moon		7 1	2			Stepping	Spaced up to the surface
7v	Scalar	3	2				Nibbling	Moderate, even, possibly due to manufacturing
	Triangular		3				Rounding	Minor on outermost edge
	Trapezoidal		2				Smoothing Polish	Even topography but minimal smoothing Small, patchy but bright areas (3); dull and patchy over the remainder; two spots of heavy, but dull polish (possible residue)
							Snap	Possible stepping limited to proximal margins
10v	Triangular			1			Nibbling	Fine, even, jagged
	Half-moon				2		Smoothing Polish	Moderate to heavy, grain levelling Heavy but patchy over surface; small bright spots just over 1mm from edge; single area near edge slightly brighter, but still matte
13v	Scalar					3	Nibbling	Mix of fine to moderate, even spacing, jagged
							Smoothing	Moderate to heavy
							Polish	Heavy, but patchy over surface; bright spots along outermost edge; three additional bright spots running vertically from edge



Polish located along non-working edge, 100x (a); patchy, preferential polish along non-working edge, 200x, 500x (b1, b2); and highly developed polish on the ventral non-working edge, 100x (c1), demonstrating isolated glossy areas (c2, 500x) and heavy, pitted polish (c3, 500x).



Low-powered Microscopy

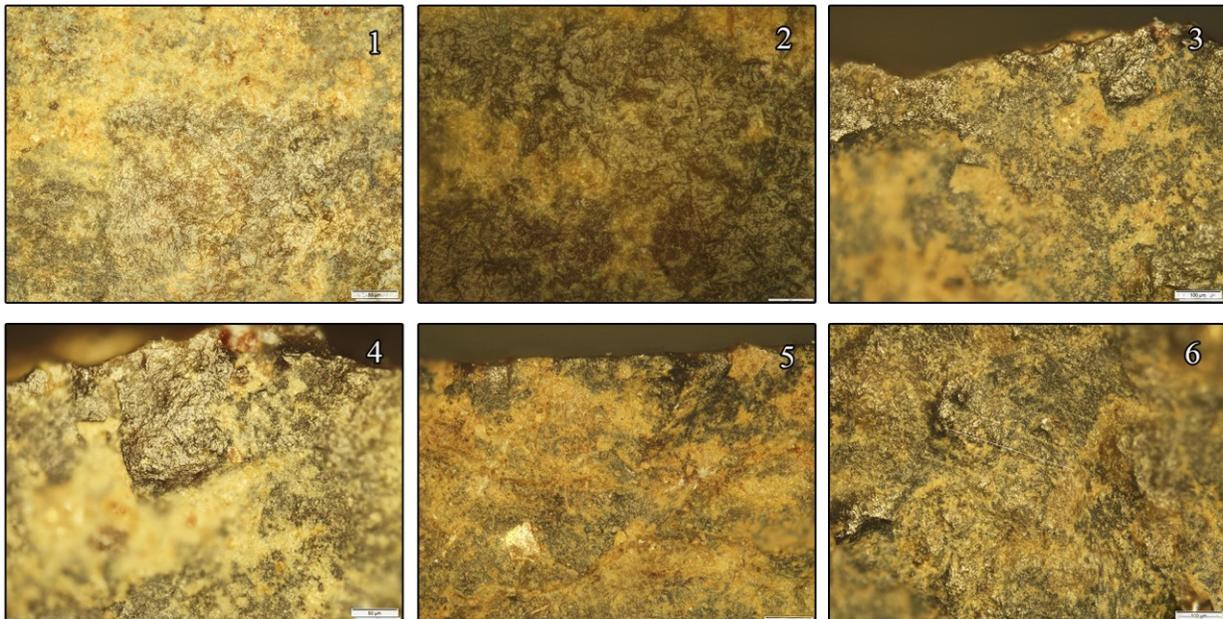


UN23 Micro-flake Analysis						Feature Analysis	
Ref	F	H	S1	S2	C	Feature	Description
1d	Scalar		3	5		Rounding	Minor, limited to outermost edge
	Triangular	2	2			Smoothing	Minor, stepping lines not smoothed over
	Trapezoidal	1	4	1		Polish	Minor, dull along central horizontal margin with single bright spot
	Irregular		1			Striations	Left oblique, very fine, near snap fracture
	Half-moon	1				Snap	One large, interior remains un-smoothed
	3d	Scalar	2				Nibbling
Triangular			1			Rounding	Light, limited to outermost edge
Trapezoidal		2				Smoothing	Minor
						Polish	Rectangular area with dull sheen on the left; uneven patch to the right; banded in two rows from edge (.5 and 1.0mm); single bright spot on upper area
						Striations	Left oblique from edge, <1mm in length
7d		Scalar	3				Smoothing
	Triangular	3	1			Polish	Dull sheen which gets heavier and more liquid-like across edge; single bright spot on upper area
	Trapezoidal	3		1		Striations	Slightly left oblique visible in dull polish
	Half-moon		1			Snap	Large, pointed, PD (?) corner broken
	10v	Scalar	5				Nibbling
						Rounding	Moderate, outermost edge

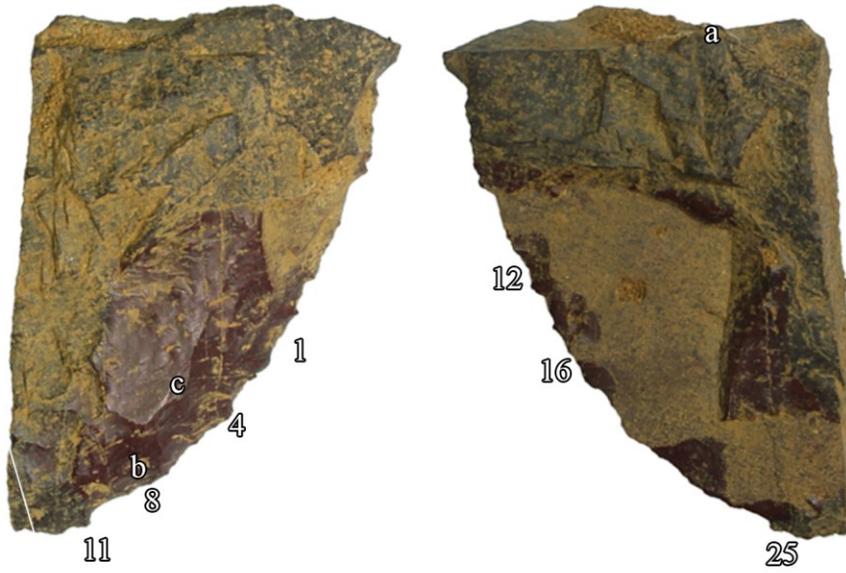
		Smoothing	Even, grain delineation
		Polish	Even, very dull
12v	No significant scarring	Rounding	Heavy rounding beside scalar scar, surface
		Smoothing	Minor to moderate, no grain delineation
		Polish	Bright spot above and parallel to the rounded area; dull and matte over remaining area; single slightly luminous area in corner of scalar scar



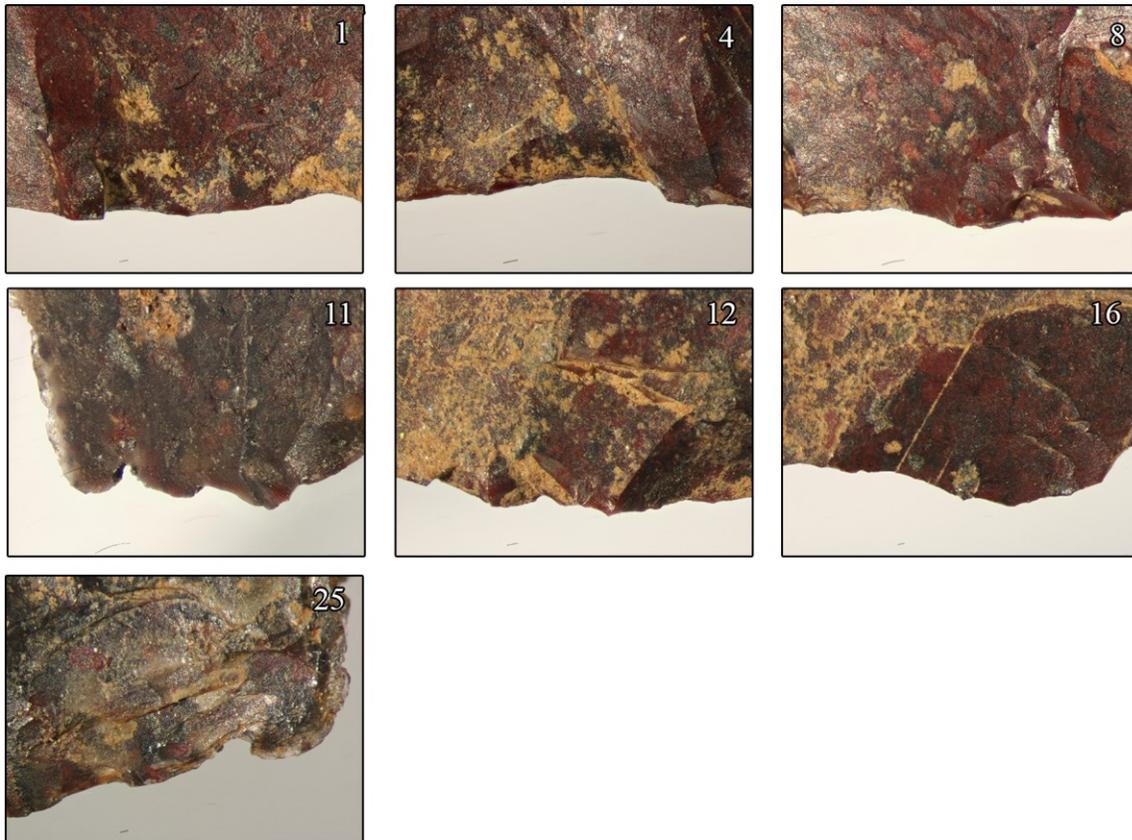
High-powered Microscopy



Heavy smoothing and translucent red residue, 100x (a); moderated rounding and smoothing of the edge within proposed hafting area, 200x (b); patchy, slightly greasy polish and minor edge rounding, 100x (c); bright, glossy polish, heavy smoothing, and faint striations (d); tentative translucent woody tissue, hyphae inter-mixed with sediments (e); heavy smoothing and even, dull polish, cellulose or collagen intermixed with sediments (f).



Low-powered Microscopy



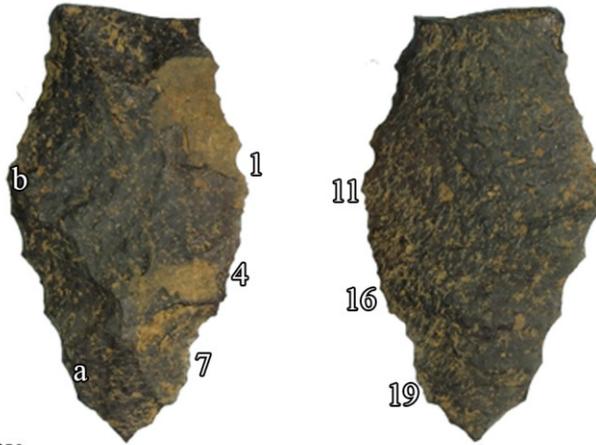
High-powered Microscopy



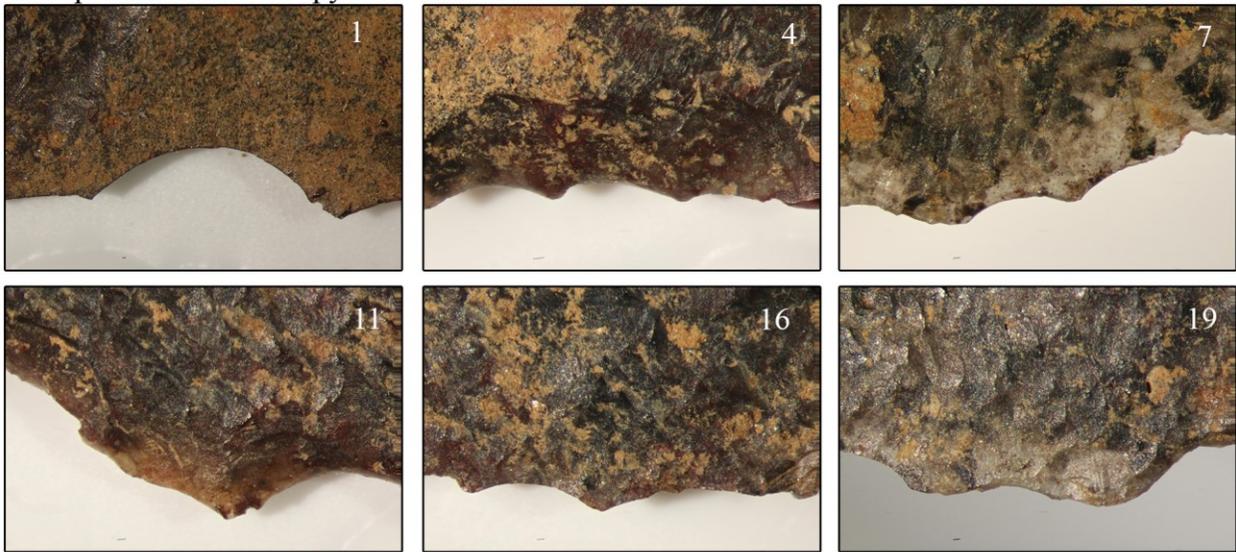
Unidentified cluster of translucent fibers within crevice on fractured plane, 100x (a); poorly developed polish and mineral inclusion, 100x (b); minor to moderate rounding and polish along scar boundaries, 100x (c).

UN25 Micro-flake Analysis						Feature Analysis	
Ref	F	H	S1	S2	C	Feature	Description
1d	Scalar			3		Nibbling	Broad, uneven; smaller, shallower nibbling within
	Triangular	2		1		Rounding	Minor on outermost edge
	Trapezoidal			3		Smoothing	Minor grain levelling of surface
						Polish	Minor, dull on surface, brighter on peaks and ridges; liquid-like on large protrusion; possible pitting near edge
						Striations	Very tentative
4d	Triangular		5			Nibbling	One right crested, broad
						Rounding	Minor
						Smoothing	Minor, no grain delineation
						Polish	Single bright spot on thickest part of edge; remainder dull polish, lessens near working edge
8d	No significant scarring					Nibbling	Broad, even; smaller and shallower within larger
						Smoothing	Moderate to heavy without grain delineation
						Polish	Bright, patchy on thicker section; almost vitreous on higher ridge that raises into a plateau; glossy in concave area
						Striations	Left oblique, very faint, many; parallel, similar
11d	No significant scarring					Nibbling	Very uneven and pointed
						Smoothing	Moderate, minor grain delineation
						Polish	Dull, liquid-like polish on slanted protrusions
						Striations	Left oblique on liquid-like polish
12v	No significant scarring					Nibbling	Uneven mixture of small to moderate, mixture of jagged and rounded edges
						Rounding	Minor on smaller nibbled areas
						Smoothing	Minor

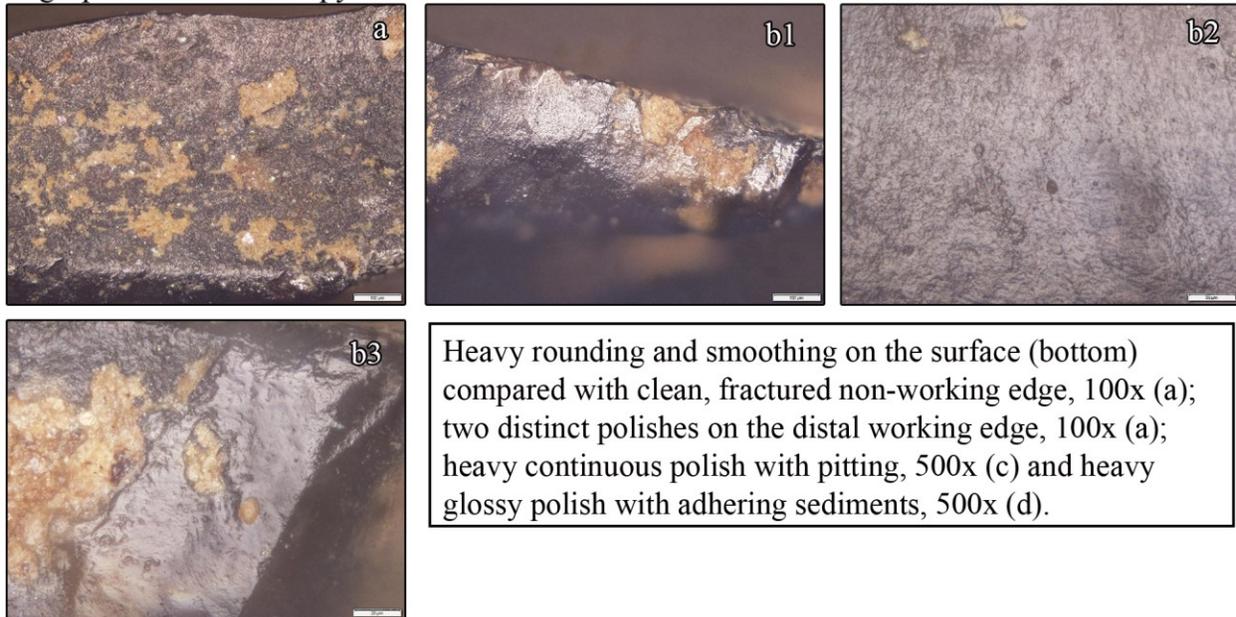
			Polish	Visible on a single jagged protrusion (highest topography)
			Striations	Tentative right oblique, lateral area from the main ridge
			Stepping	Large, probably due to manufacture
16v	Scalar	2	Nibbling	Moderate, uneven, pointed. Tentative PD damage
			Smoothing	Grain delineation present
			Polish	Sand polish, very minor, dull on all surfaces
25v			Polish	Bright spots near edge concavity (3); all closest to lateral edge, not directly on working edge
	No significant scarring		Stepping	Heavy and extensive, all hinged, 6-7 layers over 3mm section, possible due to manufacture



Low-powered Microscopy



High-powered Microscopy

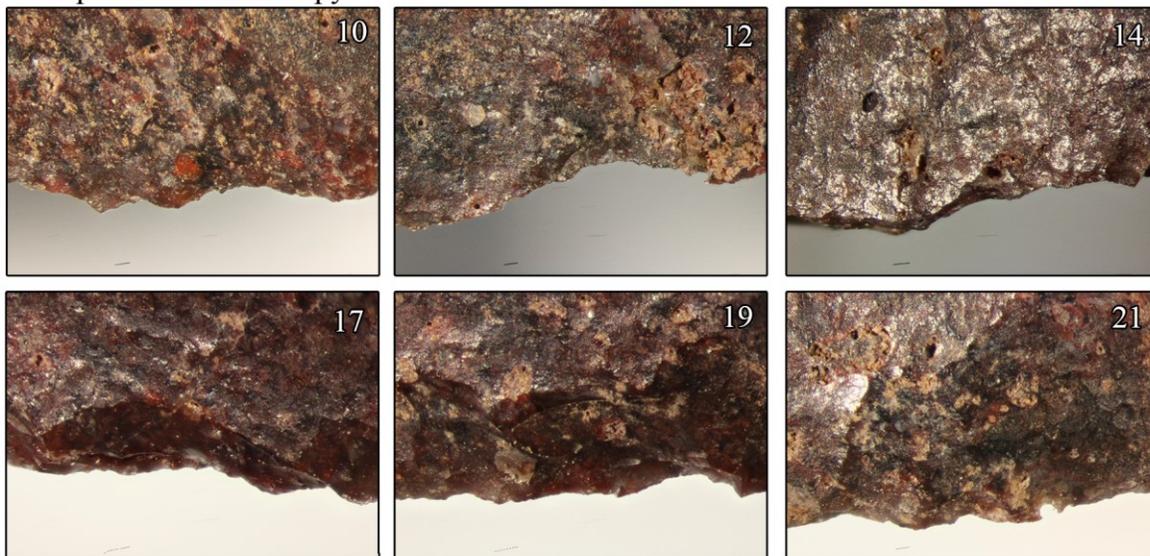


Heavy rounding and smoothing on the surface (bottom) compared with clean, fractured non-working edge, 100x (a); two distinct polishes on the distal working edge, 100x (a); heavy continuous polish with pitting, 500x (c) and heavy glossy polish with adhering sediments, 500x (d).

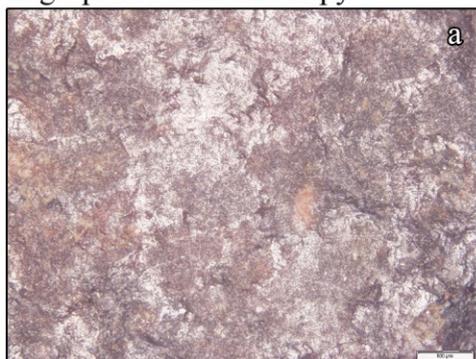
UN26 Micro-flake Analysis						Feature Analysis	
Ref	F	H	S1	S2	C	Feature	Description
1d	Half-moon				1	Snap	Single snap fracture, no scarring
4d	Triangular		3			Nibbling	Broad, left crests
	Trapezoidal		2			Rounding	Minor on outermost edge
	Half-moon				1	Smoothing	Fine to moderate, grain levelling, removal of S2 covers
7d	Scalar	3				Nibbling	Broader, 2 smaller left crested within larger
	Trapezoidal	6				Rounding	Minor to moderate
	Half-moon		1			Smoothing	Moderate, grain levelling present
						Polish	Minor, limited to highest ridges
11v	Scalar	1			1	Stepping	Very minor and smoothed over
	Triangular		3			Nibbling	Broad
	Trapezoidal		3			Smoothing	Minor, removal of S2 covers; slightly heavier near edge
	Irregular		1			Polish	Limited to thicker edge portions and peaks
	Half-moon			1			
16v	Scalar	1	1			Nibbling	Broad, lengthens to the right
	Triangular		8			Rounding	Minor
	Trapezoidal		8	3		Smoothing	Minor; S2 cover removal, grain homogeneity nearest the edge
	Irregular		3			Polish	Minor, more visible on peaks and thicker areas
19v						Stepping	Moderate, 2-4 layers, lightly smoothed and rounded
	Scalar		3			Nibbling	Broad, even
	Triangular		10			Rounding	Heavy, even
	Trapezoidal		8			Smoothing	Grain delineation present
						Polish	Minor, more visible on peaks and thicker areas



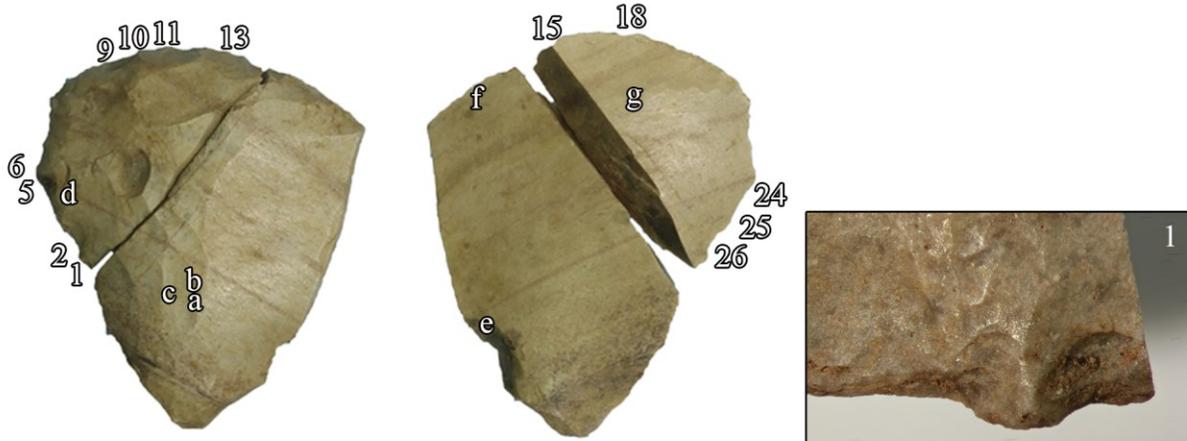
Low-powered Microscopy



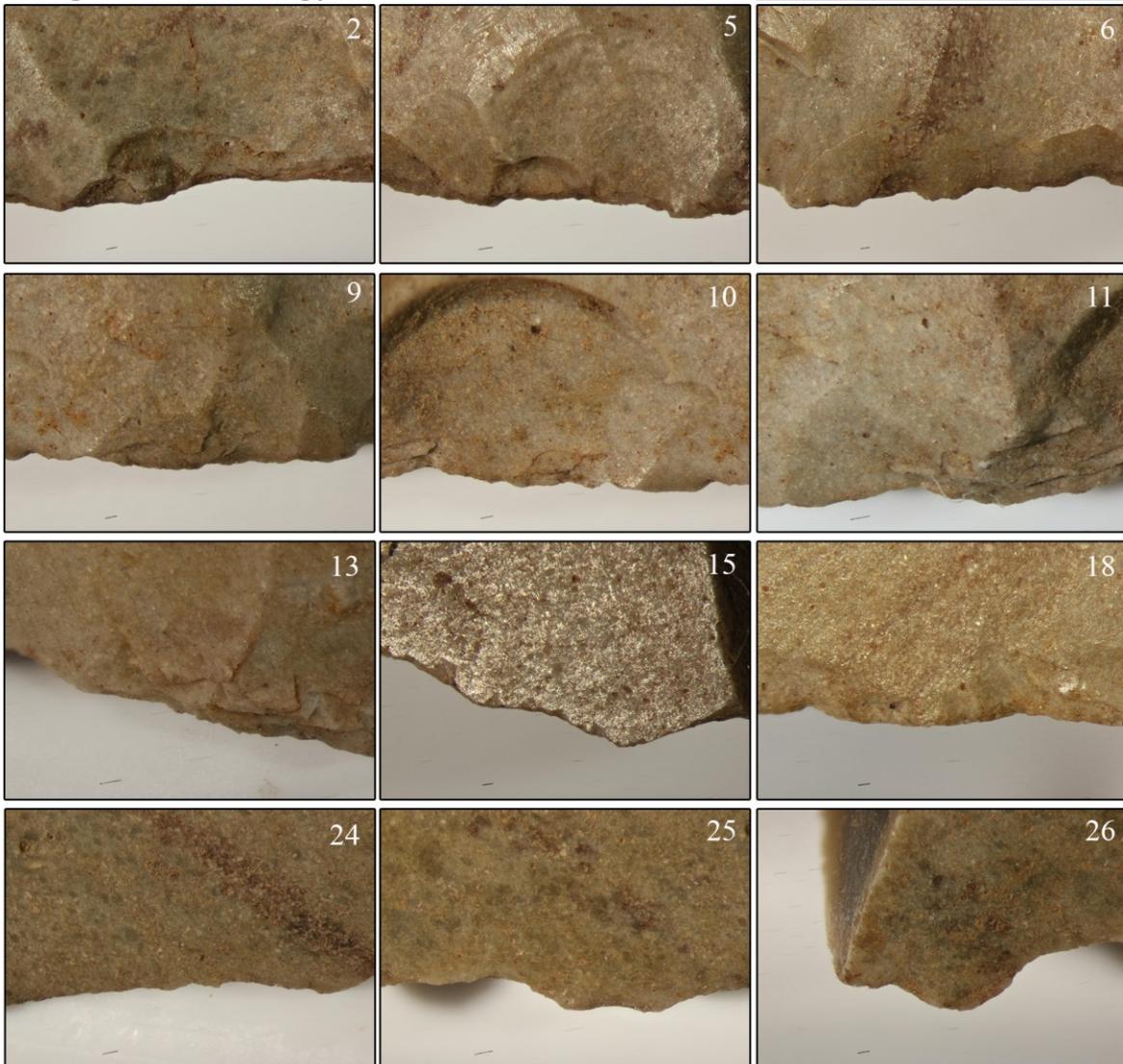
High-powered Microscopy



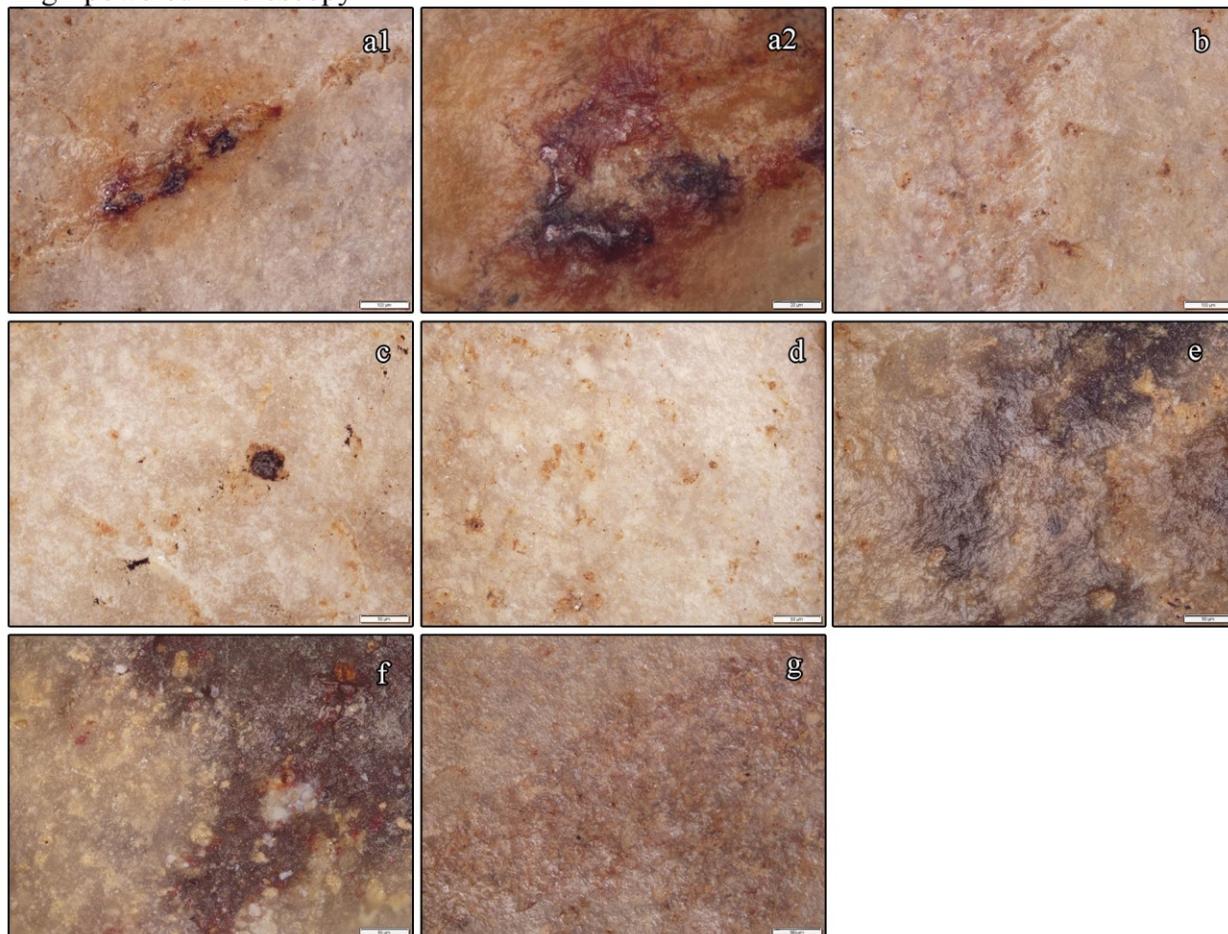
UN27 Micro-flake Analysis						Feature Analysis		
Ref		F	H	S1	S2	C	Feature	Description
10d		No significant scarring					Nibbling	Uneven, moderate to broad, sharp
							Smoothing	Very uneven, differences in grain peaks
							Polish	Minor
12d		No significant scarring					Nibbling	Fine, less sharp, within broader
							Rounding	Minor, uneven on nibbling
							Smoothing	Fairly even, grain levelling visible, complete S2 cover removal
							Polish	Very minor, limited to peaks and a single area near edge
14d	Scalar	1	1				Nibbling	Moderate
	Triangular		5	3			Rounding	Minor on thinnest section of edge
	Trapezoidal			1	1		Smoothing	Minor all over
							Polish	Heavy polish all over surface, heavier nearest edge
							Striations	Tentative very fine, within polish parallel to edge (many)
17v	Scalar		2				Nibbling	Fine on thinnest section of edge
	Triangular		2	2			Smoothing	Very minor; moderate on thicker edge
	Trapezoidal			5			Polish	Heavy on thicker edge; minor adhesive polish on surface
	Half-moon		2		2		Stepping	Single occurrence, 2 layers
19v	Scalar	1					Nibbling	Fine within broader nibbling, even
	Triangular		6				Rounding	Very minor, limited to outermost edge
	Trapezoidal		3		1		Smoothing	Minor grain levelling
							Polish	Minor adhesive polish on surface, heavy polish on thicker edge
21v	Scalar			1			Nibbling	Uneven mixture of fine to moderate, sharp
	Triangular		5				Smoothing	Minor grain delineation, heavier along edge
	Trapezoidal		4				Polish	Minor of peaks and surfaces, possibly PD
	Half-moon		1	1	2		Stepping	Minor, 2 instances with 2 layers each



Low-powered Microscopy



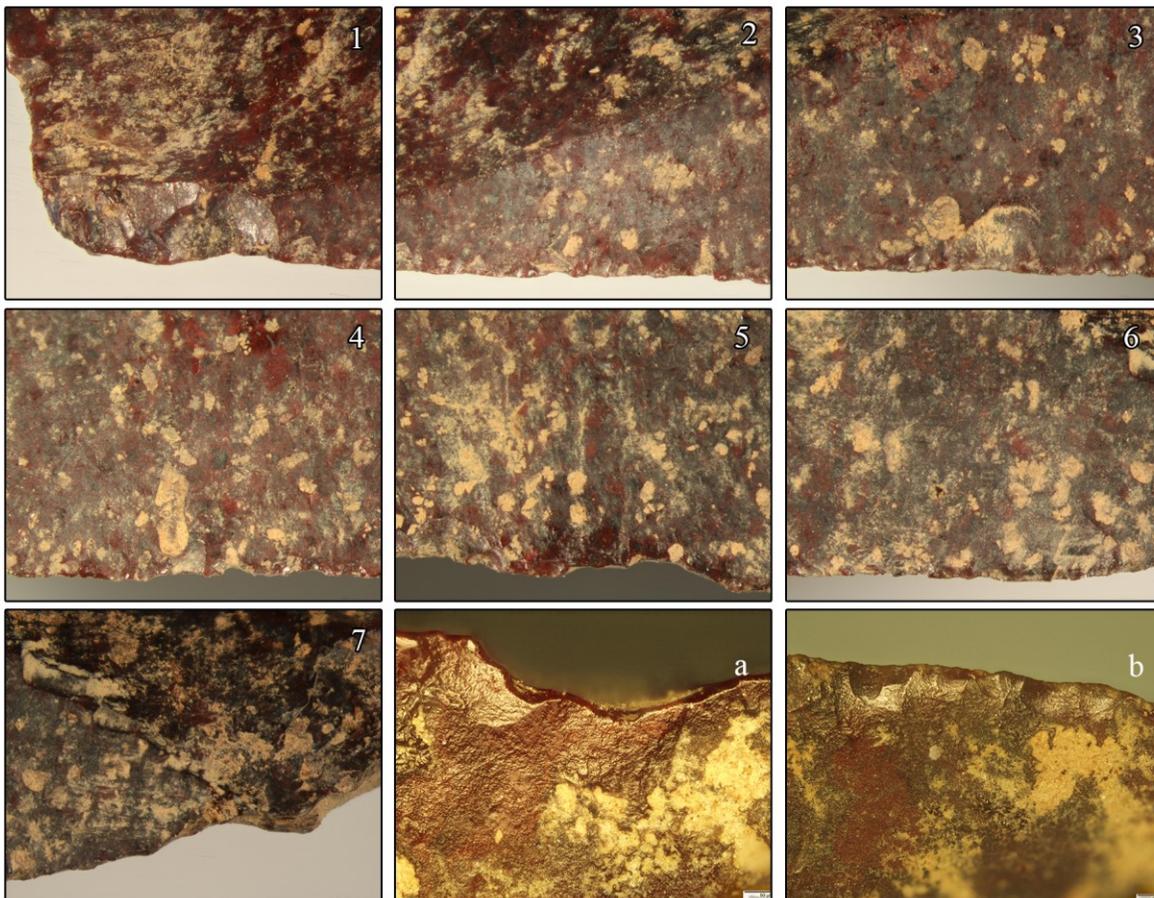
High-powered Microscopy



Dark red residue with embedded striations on central dorsal ridge, 100x and 500x (a1, a2); upper ridge showing rounding and right oblique striations, 100x (b); dark reddy black residue speck surrounded by dull polish, 200x (c); much smaller, randomly distributed black residue specks adhering to the left lateral surface, 200x (d); polish on lower portion of lateral edge, 200x (e); non-polished portion as comparison, 200x (f); even, moderately bright polish on ventral surface, 100x (g).

UN30 Micro-flake Analysis						Feature Analysis	
Ref	F	H	S1	S2	C	Feature	Description
1, 2d						Nibbling	Extremely fine, very even
						Rounding	Extensive, much thicker edge; possible grinding
						Polish	Bright, reflective on peaks and thicker portions of edge
5, 6d						Nibbling	Sharp on thin edge
9d						Nibbling	Uneven, mixture of fine and moderate
						Rounding	Very minor, limited to outermost edge
						Polish	Minor, limited to peaks and protruding areas
						Stepping	Messy, 1-3 layers, fairly minor
10d						Stepping	Minor, 2 layers, not smoothed
11d	No significant scarring					Stepping	Smoothed, 5-7 layers, large
13d						Stepping	Outermost edge very unevenly pointed, unused
15-19v						Rounding	Minor rounding of outermost edge
						Smoothing	Heavy, evenly distributed
						Polish	Heavy, even, much lighter within large scalar retouch scars
24v						Nibbling	Uneven, sharp
25v						Nibbling	Uneven, slightly rounded, moderate
						Stepping	One small area, minor
26v						Nibbling	Even, moderate

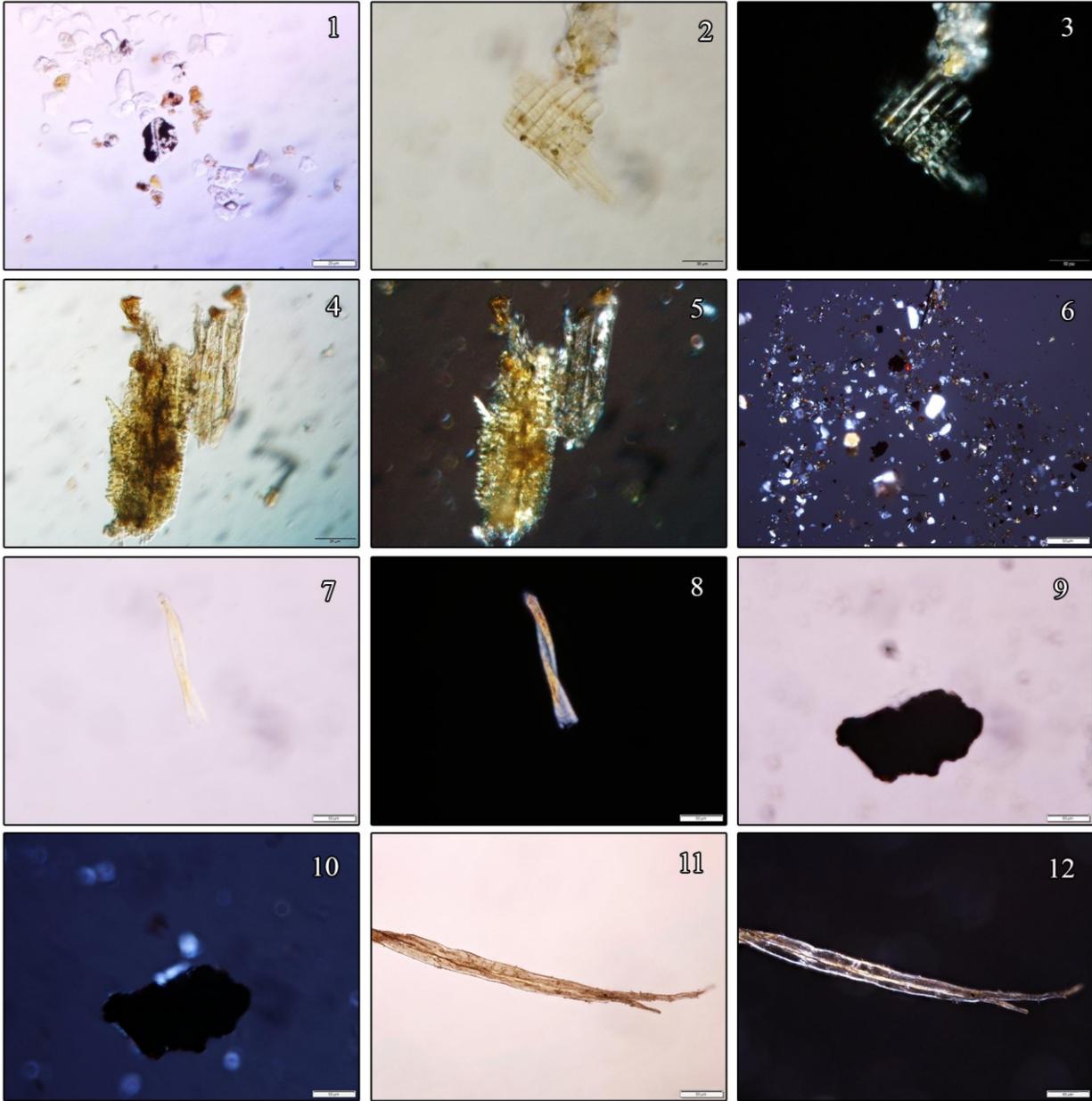
Note: while no significant use-related wears were identified, feathered and hinged scalar retouch scars were found across most of the edge.



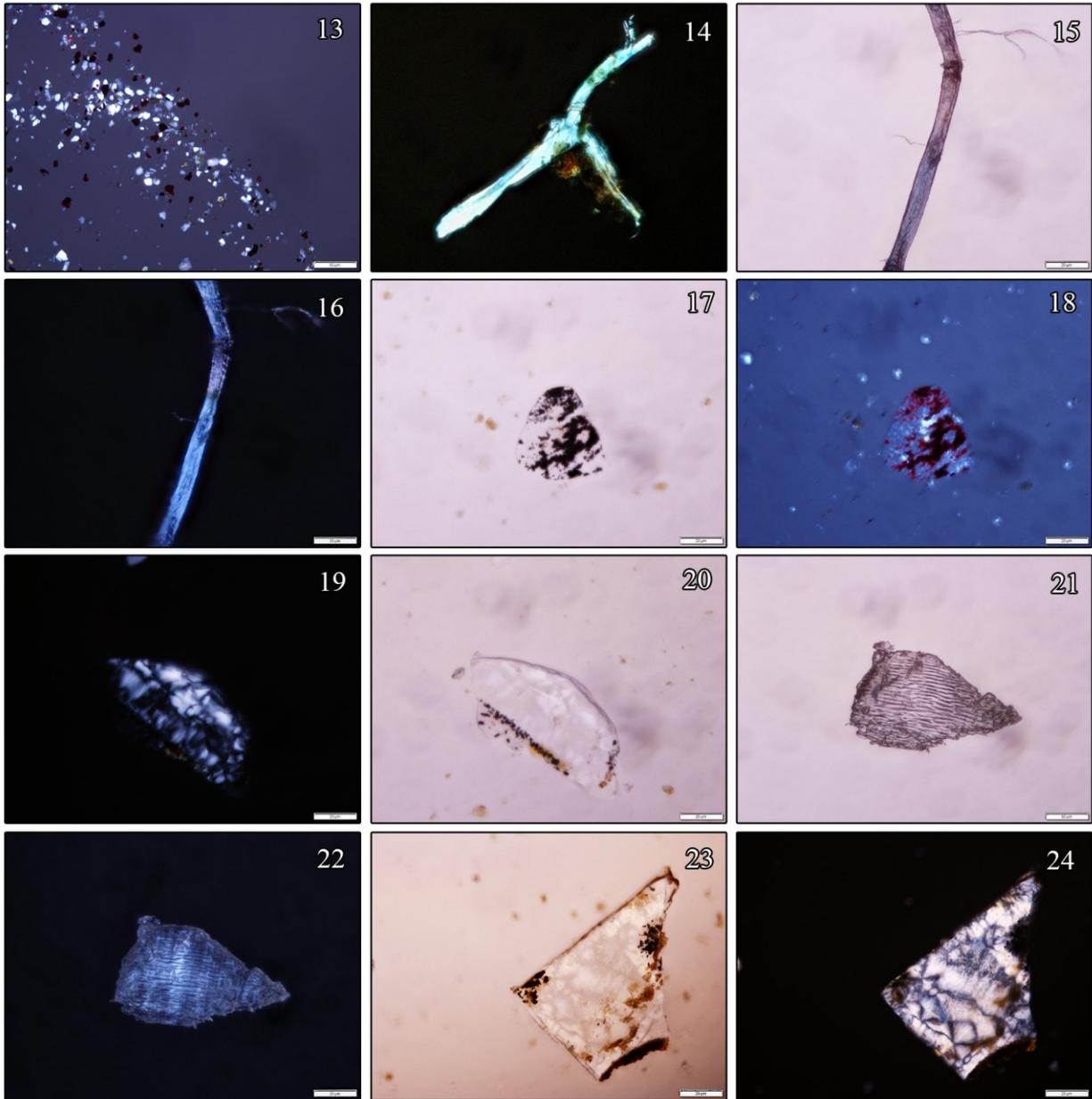
Overview photos of left lateral edge, 30x, showing heavy smoothing, rounding limited to the outermost edge, and a variety of hinged scalar, triangular, and trapezoidal scars in addition to sliced scalar scars and dull, even polish (1-7). Sliced scalar scars, rounding, and smaller areas of bright polish can be seen at 200x (a), immediately proximal of continuously distributed hinged and feathered scalar scars, 100x (b).

APPENDIX C

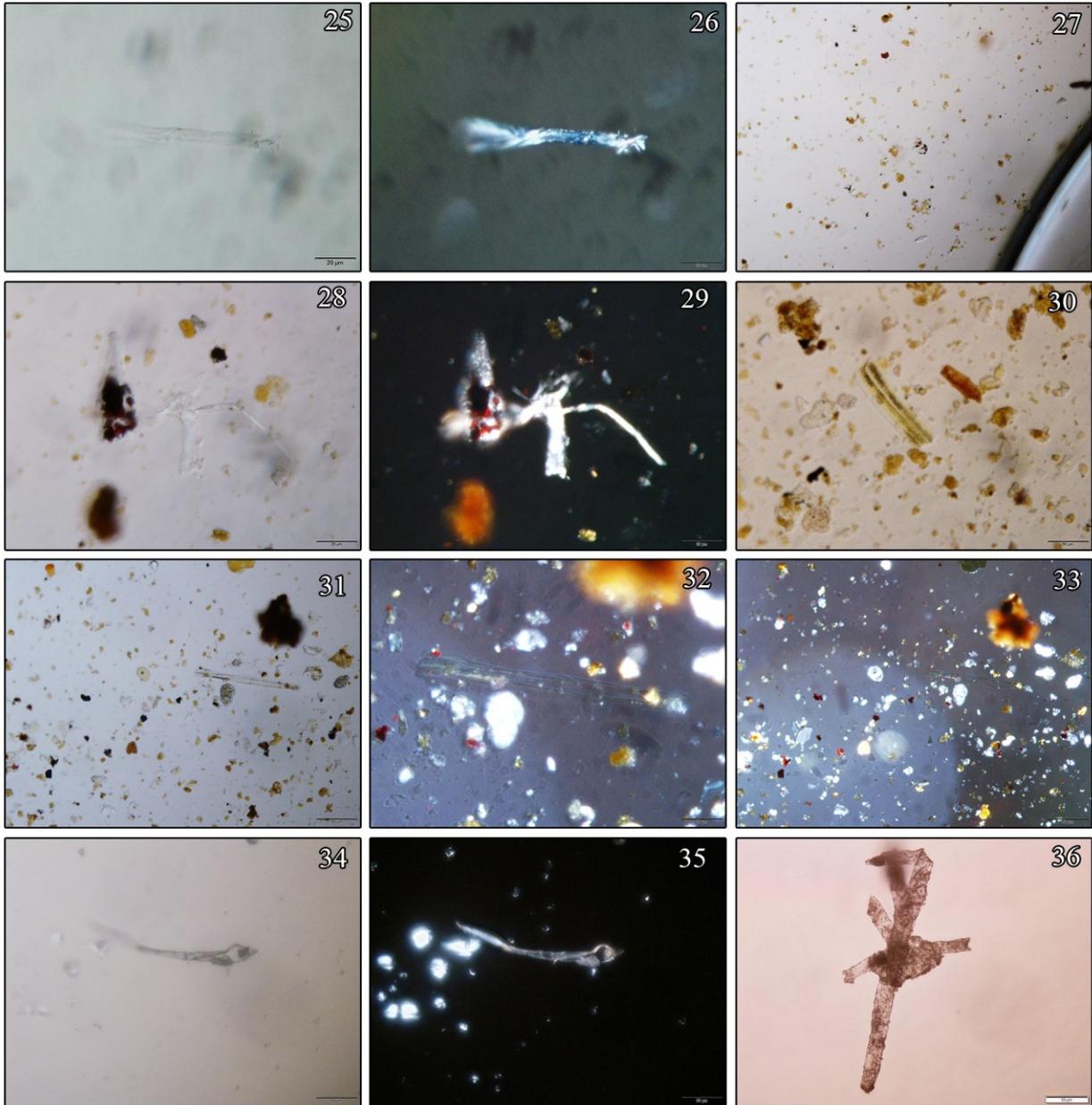
TRANSMITTED LIGHT MICROSCOPY: EXTRACTED RESIDUE IMAGES



1: Taconite flake, UN1, 500x. 2: Radial fragment of woody tissue in plane (2) and cross-polarized (3) light, UN2, 500x. 4: Damaged plant tissue in plane (4) and cross-polarized (5) light, UN2, 500x. 6: Charcoal and hematite in cross-polarized light, UN3, 200x. 7: Damaged cellulose fiber in plane (7) and cross-polarized (8) light, UN3, 200x. 9: Burnt carbon matter or charcoal in plane (9) and cross-polarized (10) light, UN4, 200x. 11: Wood fiber proper in plane (11) and cross-polarized (12) light, UN4, 200x.



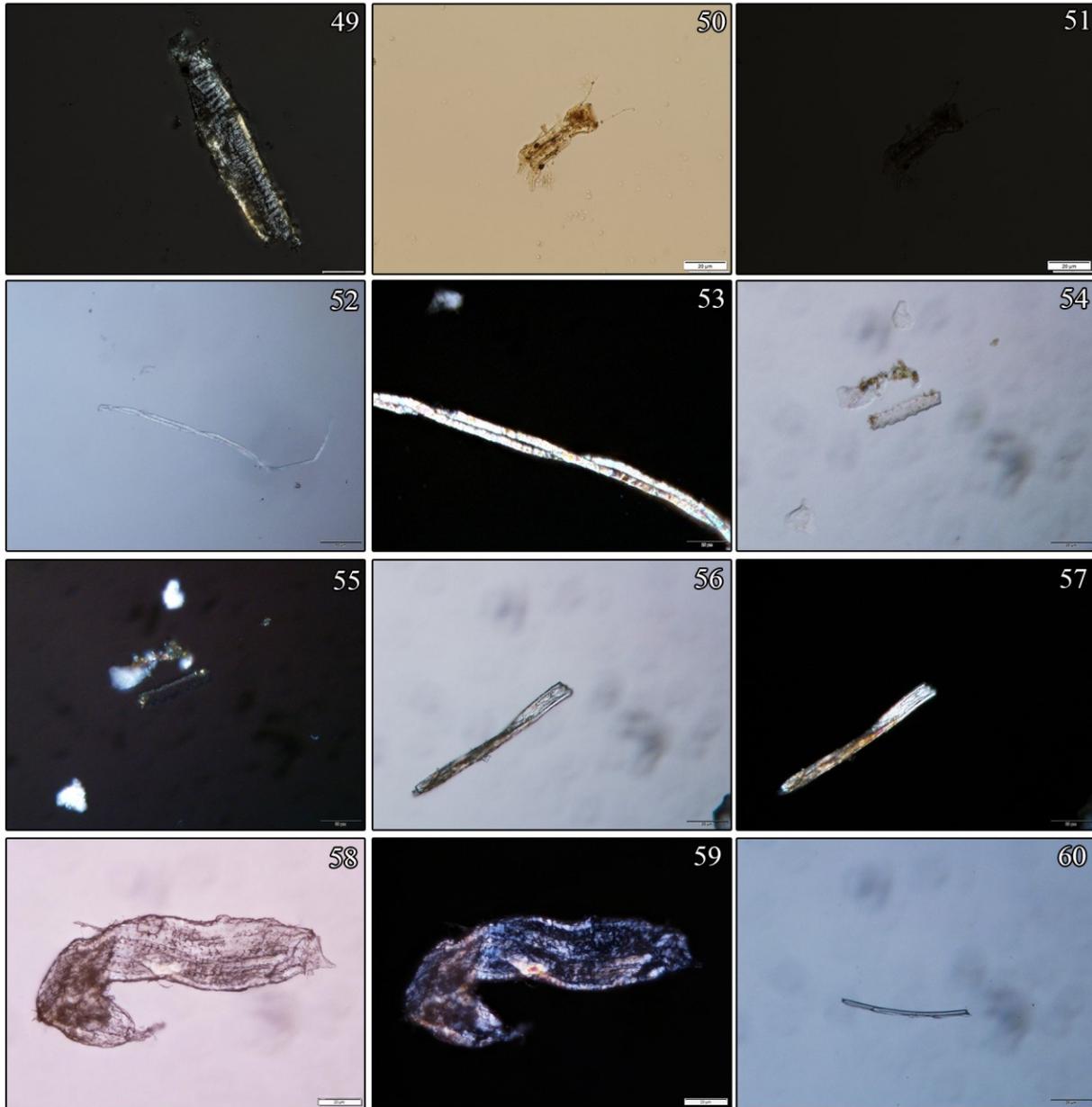
13: Hematite and charcoal in cross-polarized light, UN4, 200x. 14: Blue cellulosic fiber with adhering sediment in cross-polarized light, UN8, 500x. 15: Non-cellulosic, unidentified fiber in plane (15) and cross-polarized (16) light, UN8, 500x. 17: Taconite micro-flake in plane (17) and cross-polarized (18) light, UN9, 500x. 19: Micro-flake of Gunflint formation materials in plane (19) and cross-polarized (20) light, UN9, 500x. 21: Shell fragment in plane (21) and cross-polarized (22) light, UN9, 200x. 23: Chalcedony flake in plane (23) and cross-polarized (24) light, UN9, 500x.



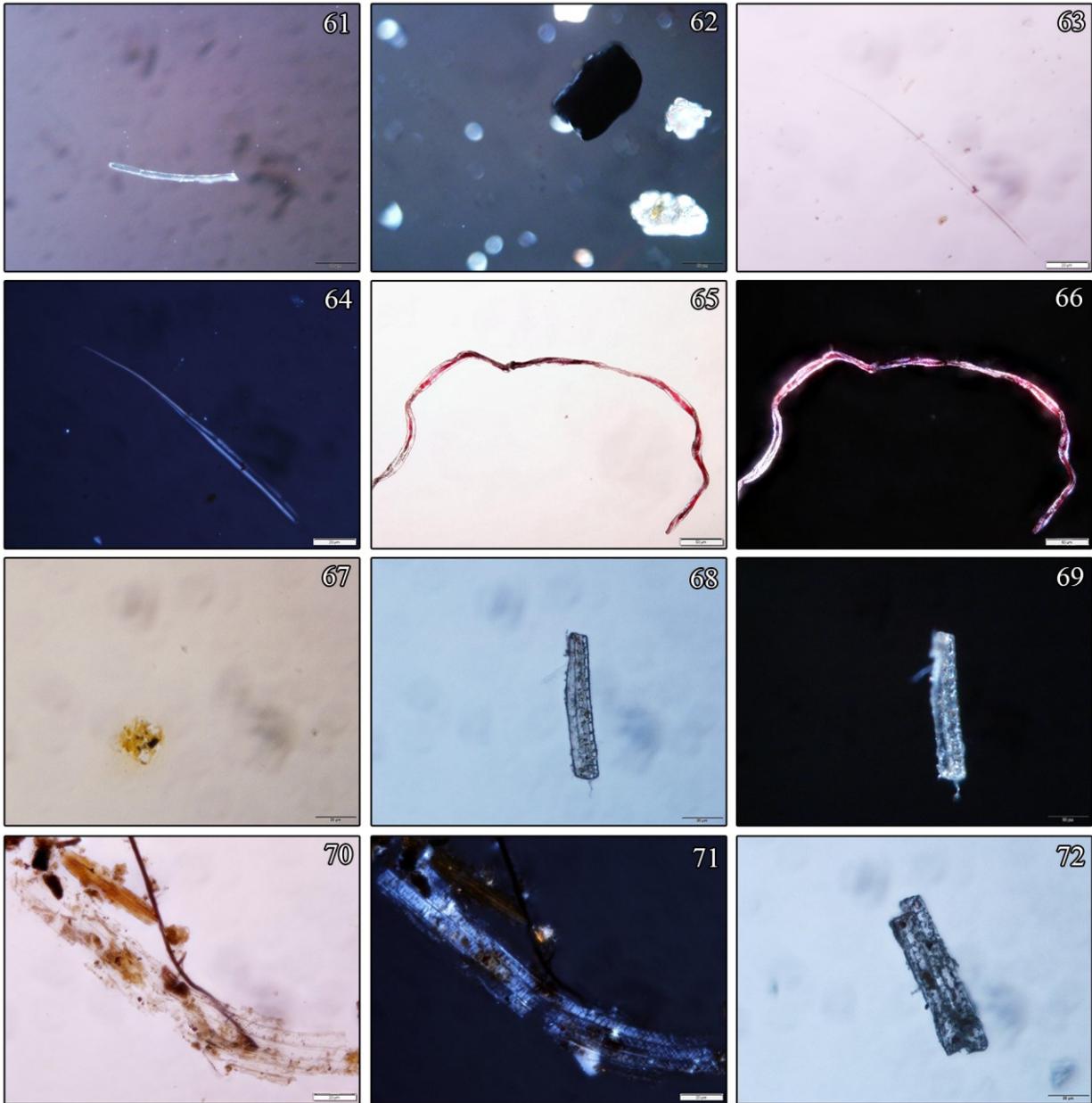
25: Tentative degraded collagen fiber in plane (25) and cross-polarized (26) light, UN10, 500x. 27: Taconite micro-flake, UN13, 100x. 28: Taconite micro-flake in association with plant fibers in plane (28) and cross-polarized (29) light, UN13, 500x. 30: Unidentified plant material, UN13, 200x. 31: Lignified cell walls of woody plant at 200x plane polarized (31) and 500x and 200x (32, 33) in cross-polarized light, UN13. 34: Trichome or diatom, likely contamination, in plane (34) and cross-polarized (35) light, UN14, 200x. 36: Unidentified material, likely plant or collagen, UN15, 200x.



37: Unidentified material, likely plant or collagen, in cross-polarized light, UN15, 200x. 38: Unidentified plant component in plane (38) and cross-polarized (39) light, UN15, 200x. 40: Fractured cellulosic fiber in plane (40) and cross-polarized (41) light, UN18, 200x. 42: Tentative pollen grain with adhering silica material, most likely contamination, in plane (42) and cross-polarized (43) light, UN18, 200x. 44: Degraded fragment of feather barbule in plane (44) and cross-polarized (45) light, UN19, 500x. 46: Fragmented plant fiber in plane (46) and cross-polarized (47) light, UN21, 500x. 48: Unidentified plant fiber, UN21, 500x.



49: Unidentified plant fiber in cross-polarized light, UN21, 500x. 50: Damaged bilobate phytolith in plane (50) and cross-polarized (51) light, UN21, 500x. 52: Very fine, unidentified fiber, in plane (52, 200x) and cross-polarized (53, 500x) light. 54: Wavy-edge phytolith in plane (54) and cross-polarized (55) light, UN23, 500x. 56: Possible degraded phytolith in plane (56) and cross-polarized (57) light, UN24, 500x. 58: Plant material in plane (58) and cross-polarized (59) light, UN25, 500x. 60: Chiton fragment, UN25, 500x.



61: Chiton fragment in cross-polarized light, UN25, 500x. 62: Charcoal in cross-polarized light, UN25, 200x. 63: Whisker raphide in plane (63) and cross-polarized (64) light, UN26, 500x. 65: Degraded, dyed cellulosic fiber, contamination, in plane (65) and cross-polarized (66) light, UN26, 200x. 67: Yellow iron oxides, UN27, 500x. 68: Woody paranchyma cell in plane (68) and cross-polarized (69) light, UN27, 500x. 70: Higher plant material in plane (70) and cross-polarized (71) light, UN27, 500x. 72: Woody paranchyma cell, UN29, 500x.



73: Woody paranchyma cell in cross-polarized light, UN29, 500x. 74: Chert micro-flake with microscopic edge damage, a feather termination, and striations in plane (74) and cross-polarized (75) light, UN30, 200x. 76: Heavily damaged collagenous structure in plane (76) and cross-polarized (77) light, UN30, 500x. 78: Taconite micro-flake, UN33, 200x. 79: Unidentified fiber, UN33, 200x. 80: Carbonized plant matter in cross-polarized (80), plane (81), and incident (82) light, UN33, 200x. 83: Unidentified organic structure in plane (83) and cross-polarized (84) light, UN33, 500x.

APPENDIX D: TOOL METRICS

ID	Context	Depth (cm)	Measurements* (mm)	Edge Angle (°)	Type
UN1	489N/534E-SEQ	0-5	45.4/42.6/11.0	25-30	Expedient
UN2	490N/534E-NEQ	0-5	47.7/28.8/7.9	42-44	Expedient
UN3	495N/533E-SE	20-25	28.2/77.4/15.8	60-65; 55-60	Informal
UN4	496N/529E-SE	5-10	17.0/21.6/10.0	62-80	Informal
UN6	502N/536E-SW	20-25	25.8/21.3/3.4	65-70; 35	Informal
UN8	504N/547E-NE	115-120	30.5/29.1/7.0	40-45	Informal
UN9	505N/504E-SW	10-15	32.3/26.5/5.1	50-55	Informal
UN10	505N/519E-SE	15-20	25.8/22.8/9.36	80-90; 65-70	Formal
UN11	505N/546E-SW	100-105	73.8/36.5/10.8	55-60	Expedient
UN13	507N/546E-SW	25-40	63.1/46.1/16.6	60-65	Informal
UN14	509N/518E-SE	15-20	34.2/29.0/11.1	80-90	Informal
UN15	509N/529E-NW	5-10	81.3/31.3/12.9	50, 70	Expedient
UN18	509N/545E-SE	15-20	28.0/20.1/4.4	60-65	Informal
UN19	512N/522E-NE	15-20	46.8/30.7/8.3	55-60; 65	Informal
UN21	514N/521E-NE	15-20	33.3/28.4/10.1	60-65; 50-55	Formal
UN23	516N/540E-NE	10-15	25.9/10.6/6.2	65-70	Formal
UN24	516N/544E-NE	20-25	80.3/24.3/9.3	40-50	Informal
UN25	517N/540E-NW	20-25	49.8/36.1/14.5	47-53	Informal
UN26	518N/539E-SE	15-20	108.5/49.8/21.1	25-35	Informal
UN27	518N/539E-SW	5-10	41.6/23.2/2.7	30-40	Expedient
UN30	526N/542E-SE	0-5	34.5/38.8/7.0	45	Formal
UN33	512N/525E-NE	25-30	72.7/52.5/7.6	20; 45	Formal

*Measurement were recorded as length/width/thickness. All measurement were recorded with digital calipers and have been rounded to the nearest decimal point.

Note: All tool within the sample excluding UN6 and UN30 consisted of varieties of taconite. UN6 and UN30 are fine-grained banded gunflint formation cherts.

APPENDIX E: SUMMARY OF RESULTS

	Wear	IR	ER	BT	AS	GC/MS	Interpretation
UN1	Push-pull motion Bidirectional Longitudinal Medium material Fresh wood or bone Unhafted	N/A	Taconite microflake	Starch	Positive	Hexadecanoic acid Octadecanoic acid Myristic acid	Expedient Unhafted Bone spokeshave
UN2	Pulling motion Unidirectional Longitudinal Soft or woody plant Unhafted	N/A	Woody tissue, damaged plant material	N/A	N/A	Octadecanoic acid Hexadecanoic acid Heptadecane Tridecane Pentadecane Azealic acid	Expedient Multi-purpose handheld knife
UN3	Pull motion Unidirectional Longitudinal Soft plant Hafted	N/A	Charcoal, hematite, damaged cellulosic fiber	N/A	N/A	Octadecanoic acid Cis-9-octadecanoic acid Trans-9-hexadecanoic acid Myristic acid Dodecanoic acid Propanoic acid (lactic)	Informal Male hafting Multi-purpose scraper; root and animal processing
UN4	Push-pull motion Bidirectional Longitudinal Medium to hard material Seasoned wood and bone Hafted	N/A	Wood fiber, hematite, charcoal, burnt carbon matter	N/A	N/A	Dioxa-disilaoctaine	Informal Male hafting Wood planer
UN6	Push-pull motion Unidirectional Longitudinal Dry hide Hafted	N/A	N/A	Carb	Positive	N/A	Informal Juxtaposed hafting Dry hide scraper
UN8	Cutting motion Bidirectional Transverse Medium-hard material Seasoned wood Unhafted	N/A	Plant matter, non-cellulosic fiber	N/A	N/A	Propanoic acid Octadecanoic acid Hexadecanoic acid Trans-9-hexadecanoic acid Benzene Tetradecanoic acid Nonanoic acid Propanoic acid (lactic)	Informal Handheld Knife
UN9	Pulling motion Unidirectional Longitudinal Medium-hard Hard wood Hafted	N/A	Taconite microflake, flake with charcoal or magnetite, shell	Fatty acids	Positive	Octadecanoic acid Hexadecanoic acid Benzaldehyde Pentadecanoic acid Hexadecanoic acid	Informal Male or juxtaposed hafting Wood planer

UN10	Push-pull motion Bidirectional Longitudinal Medium material Fresh bone Hafted	White, amorphous residue under matrix	Twisted, degraded collagen	N/A	N/A	Borate	Formal Male hafting Bone planer
UN11	Cutting motion Bidirectional Transverse Soft and medium material Fresh bone and meat Unhafted	N/A	N/A	N/A	N/A	Octadecanoic acid Hexadecanoic acid Benzene Tetradecanoic acid Dodecanoic acid Octadecenyonic acid Propanoic acid Glyoxylic acid, di-TMS	Expedient Handheld Multi-purpose knife
UN13	Pulling motion Unidirectional Longitudinal Medium, medium-hard Fresh bone or wood Unhafted	N/A	Taconite flake, taconite with plant fibers, woody plant lignin	N/A	N/A	Octadecanoic acid Palmitelaidic acid Benzene Tetradecanoic acid Dodecanoic acid Hexadecanoic acid Octanoic acid Propanoic acid Glyoxylic acid, di-TMS	Informal Handheld Multi-purpose scraper
UN14	Pulling motion Unidirectional Longitudinal Soft and medium-hard Fresh bone, meat Hafted	Embedded fibers in hafting area	N/A	Fatty acids, starch	Positive fatty acids	Glyoxylic acid, di-TMS Dodecanoic acid Trimethylsilyl ether of glycerol	Informal Male hafting Multi-purpose scraper
UN15	Pull-cut motion Unidirectional Transverse Soft and medium-hard Fresh bone, meat Unhafted	N/A	Plant material, charcoal and hematite	Fatty acids, carb.	Positive fatty acids, carb.	Propanoic acid Dimethylsilyloxy-tridecane	Expedient Handheld Multi-purpose knife
UN18	Cutting motion Bidirectional Transverse Soft and medium-hard Fresh bone, meat Hafted	Fibers embedded in matrix	Degraded cellulosic fiber	Fatty acids	Positive	Hexadecanoic acid Oleanolic acid Methanone Dihydroxanthin Octadecanoic acid Palmitelaidic acid Dodecanoic acid Octadecanoic acid Propanoic acid	Informal Male hafting Butchering knife
UN19	Pulling motion Unidirectional Longitudinal Soft material Fresh hide, soft plant Hafted	N/A	Degraded feather barbule fragment	N/A	N/A	Glyoxylic acid, di-TMS	Informal Male hafting Multi-purpose scraper

UN21	Pull-cut motion Unidirectional Transverse Plant or fibrous material Hafted	Amorphous fatty and bone residue (white amorphous and crystalline)	Plant fiber, collagen, bilobate phytolith	N/A	N/A	Glyoxylic acid, di-TMS	Formal Male hafting Whittler, multiple materials
UN23	Pulling motion Unidirectional Longitudinal Fresh hide Unhafted	N/A	Wavy-edged phytolith; very fine fiber	Fatty acids	Positive	N/A	Formal Handheld Fresh hide scraper
UN24	Pulling motion Unidirectional Longitudinal Medium-hard Dry wood, bone Hafted	Translucent red residue, opaque white residue, longitudinally striated muscle or epidermal woody cells	Degraded phytolith	Fatty acids	Positive	Glyoxylic acid, di-TMS	Informal Wrapped hafting Wood, woody plant scraper
UN25	Pull-cut motion Unidirectional Transverse Soft materials Plant and meat Unhafted	N/A	Plant material	Carb.	Positive	Octadecanol Dodecanoic acid Ethanedioic acid	Informal Handheld Multi-purpose knife
UN26	Pull-cut Unidirectional Transverse Medium-hard material Dry wood, dry bone Unhafted	N/A	Whisker raphide, cellulosic fiber	Carb.	Positive	Glyoxylic acid, di-TMS	Informal Handheld Multi-purpose knife
UN27	Pushing motion Unidirectional Longitudinal Medium-hard material Dry bone, wood Unhafted	N/A	Plant material, yellow pigment, woody cell	Fatty acids	Positive	Ethanedioic acid Benzene	Expedient Handheld Wood planer or scraper
UN30	Pushing motion Unidirectional Longitudinal Dry hide Hafted	Hafting resin with striations	Chert microflake with wear, unidentified organic structure	Fatty acids, starch	Positive fatty acid	N/A	Formal Juxtaposed hafting Dry hide scraper

UN33	Hafted	N/A	Taconite microflake, carbonized plant matter, phytolith	N/A	N/A	Hexadecanoic acid Ethanedioic acid Gluconic acid Octanoic acid Dimethyltrioxa- silatetradecanol Glyoxylic acid, di-TMS	Formal Juxtaposed hafting Inconclusive
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