CHAPTER 5

RESIDUE ANALYSIS

5.1 Residue Analysis Methods and Procedures

Using various chemical reagents, it is sometimes possible to detect the presence of minute amounts of organic residues (i.e., blood, fats, oils, resins, plant constituents) adhering to stone tools (see Briuer 1976; Broderick 1979,1984; Anderson 1980; Coughlin and Classen 1982; Loy 1983,1987; Loy and Nelson 1987; Shafer and Holloway 1979; Paull 1984; Fullagar 1986; Deal and Silk 1987; Gurfinkel 1987; Gurfinkel and Franklin 1988). Such analyses assume that there is a transferrence of plant and/or animal matter onto the surface and/or into interstices between crystalline grains of a stone tool during use, and that these deposits may persist as a residue for extended periods of time.

In the present study, tests to determine the possible presence of blood, starch, and plant lignin were performed following procedures outlined by Loy (1983), Broderick (1984), and Paull (1984). These tests were undertaken to generate data that might indicate whether the majority of key-shaped formed unifaces were used primarily on contact materials derived exclusively from animals (i.e., fresh bone), from plants (i.e., bark or wood), or both.

The first step in the analysis was to identify potential residue deposits adhering to the concave or "opposite" margins of key-shaped formed unifaces using a Nikon stereoscopic light microscope at magnifications ranging between 6.4 X and 40 X. Fortunately, the steep retouch along these margins is characterized by a preponderance of step- and hinge-terminated flake scars that are conducive to trapping and retaining residue deposits (see Chapter 5.2 and Figure 66).

During examination of the 129 prehistoric tools, residues were observed on 37 items, constituting 28.7% of the sample assemblage. It is possible that many more items initially bore residue deposits, however, some depositional contexts or recent artifact washing or brushing may have removed them.

Eighteen specimens bearing sufficient (i.e., 15 to 30 mg) residue deposits were judgementally selected and subjected to three separate reagent tests: (1) the "Hemastix" test for blood haemoglobin (Loy 1983); (2) the Iodine test for starch and plant parts; and (3) the Phloroglucinol-Hydrochloric acid test for plant lignin (Broderick 1984; Paull 1984). These simple "spot" tests were chosen because they are sensitive, relatively inexpensive, and can be performed quickly and safely with standard laboratory apparatus.

Alternate residue analysis methods such as the benzidine test for blood (Hawk *et al* 1948; Lee 1982; Broderick 1984; Paull 1984) and Sudan III and IV tests for fatty residue (Johansen 1940:63; Broderick 1983; Paull 1983) were not

undertaken in this study. The benzidine test duplicates the "Hemastix" test and involves carcinogenic agents. The Sudan III and IV tests do not distinguish between plant-derived and animal-derived fatty residues, and there is some possibility of obtaining spurious results caused by fats and oils being tranferred to tool surfaces because of artifact handling during washing, cataloging, and analysis.

5.1.1 "Hemastix" Test for Blood Haemoglobin

The Hemastix test strip was devised by Ames Division, Miles Laboratories Ltd. to determine the presence of blood in urine. The strips are extremely sensitive to haemoglobin and can detect concentrations between 5000 and 20,000 molecules in one milliliter of fluid. A single mammalian red corpuscle contains approximately 70 million haemoglobin molecules, therefore, this test is considered to be an extremely sensitive indicator for blood (Loy 1983; Paull 1984).

Loy (1983,1987) is confident that blood haemoglobin can survive extended periods of time under optimum conditions. Items examined in the present study are less than 4000 years old, and most have been buried in dry silty soils. Consequently, I reasoned that if the primary function of these items involved working fresh animal bone having bits of flesh adhering, some traces of blood should be present and detectable on at least a few tools.

The test is quick, simple, and involves only the Hemastix, physiological saline solution, and the specimens to be analyzed. First, a Hemastix is dampened with physiological saline solution (distilled water and NaCl) and placed on a slide under a binocular microscope at a magnification between 10 X and 20 X. A drop of saline solution is then placed on the functional edge or surface of the tool with a glass pipette. Next, a sample of residue deposits adhering to the tool edge/surface are scraped off and agitated within the drop of saline solution with the end of the pipette and allowed to sit for between four and six minutes. The residue sample solution is then removed with a pipette, placed on the dampened Hemastix, and examined under 40 X magnification. A weak positive reaction is indicated by small flecks of green appearing over several areas of the Hemastix pad, a moderate reaction by patchy areas of medium or dark green over several areas of the pad, and a strong reaction by turning the entire pad dark green.

False reactions for the Hemastix test can be obtained by fresh fruit or vegetable peroxidases, however, peroxidases are quickly degraded and are not a concern for archaeological specimens (Broderick 1984:4). Loy (1983) also indicates that chlorophyll can produce a spurious positive reaction, which might possibly account for some weak reactions obtained in this study (see Chapter 5.2.1). Copper oxides also invoke a false positive result (Paull 1984:247), although they are not common in most soil matrices. Any tools suspected of being associated with copper compounds should be eliminated from testing. Also, residues should *not* be removed from the sample artifacts with metal implements (i.e., such as a stencil knife as suggested by Paull [1984]) to avoid a possible false reaction caused by small fragments of metal that might be dislodged into the sample solution.

5.1.2 Iodine Test for Plant Starch

Aqueous iodine (I-), is a reagent that reacts by turning starch a blue-black colour. Starch is present in many plant cells, particularly storage cells within roots, corms, bark, and stems (Esau 1953:27-28). Approximately 20% to 25% of starch is comprised of amylose, which is water soluable; the remaining 75% to 80% consists of non-soluable amylopectin (Morrison and Boyd 1967:1027). The latter may be preserved under certain conditions in the archaeological record (Broderick 1984).

The reagent is prepared by disolving 2.0 gr of Iodine (I) and 500 mg of Potassium Iodide (KI) into a mixture of 40 ml of distilled water and 20 ml of ethanol. The testing procedure is outlined in Broderick (1984) and Paull (1984). First, a small sample of residue (5 to 10 mg) is scraped from the functional edge of the tool onto a microslide at 10 X to 20 X magnification. Next, a drop of reagent is placed on the residue sample and magnification is increased to 40 X. A positive reaction is recorded if obvious organic constituents in the sample (i.e., tiny translucent particles or detritus which move freely or concentrate at the edge of the reagent drop) turn light or dark blue, violet, or black.

False positive results for the Iodine test, as well as for the Phloroglucinol-HCl Acid test (below), can also be obtained for plant parts (i.e., rootlets, decayed wood) that are a natural and incidental component of many soil matrices. Artifacts buried in such contexts might conceivably accumulate these plant parts on their surfaces or within deep flake scars. However, I argue that in the absence of a naturally occurring medium that could firmly bond or adhere these parts to the tool (e.g., calcium carbonate or ferrous precipitates), they would be very easily removed by even the slightest washing or brushing. The possibility of non-culturally derived plant part contamination was acknowledged during this study, and care was taken to ensure that this agency did not lead to misleading conclusive results. A further discussion of this potential source of error is presented below in Chapter 5.3.

5.1.3 Phloroglucinol-HCl Acid Test for Plant Lignin

Phloroglucinol saturated in concentrated Hydrochloric acid (HCl) is a reagent that reacts with lignin in plant parts, turning them reddish-purple, magenta, or red. Lignin is a durable organic substance related to cellulose, and is a component of woody plant cell walls and the cementing materials between them. The testing procedure used in this study is outlined by Paull (1984). First, a small sample of residue (5 to 10 mg) is scraped from the functional tool edge onto a microslide with a stencil knife under a magnification of between 10 X and 20 X. Next, a drop of reagent is placed on the sample, and magnification increased to 40 X. A positive reaction is indicated if any constituents of the residue sample resembling plant parts/detritus or woody fibres turn light reddish-purple, magenta, or red.

Because interpretations of the residue analyses results were subjective in nature (i.e., they relied on visual assessments of colour and quantity) and thus researcher bias was possible, several witnesses were asked to provide verification for "strong" or "moderate" reaction results. Dr. Erle Nelson, Laurie Milne, Ian Kuijt, and Chris Knusel observed many of the reactions obtained during the haeomoglobin test; Murielle Nagy and Luke Dalabonna witnessed "strong" positive results obtained for the plant lignin test; and Ian Kuijt, Chris Knusel, and Dr. Jon Driver verified "strong" and "moderate" results of the starch test.

5.2 Residue Analysis Results

5.2.1 Results of the "Hemastix" Test

Of the eighteen tools tested for blood (haemoglobin), only three produced positive reaction results (Table 3). Two tools from the Mid-Fraser River region (EeRk 4:19-2119 and EeRl 7:1000) indicated weak positive reactions. Only one tool (FiRs 1:5699 from Punchaw Lake), indicated a strong positive reaction. Indeed, dark reddish brown blotches considered typical of blood residues (Nelson, pers. comm. 1987) are evident on the functional edge and dorsal surface of this tool. The distal half of this tool's distal projection is absent, and it is possible that it might have been used for processing animal-derived materials after it had broken. Recycling of broken or worn tools was not an uncommon practice on the Plateau. Alternately, human blood may have been deposited on this tool when the distal projection snapped off during use, resulting in its user being cut (see also Chapter 7.7).

The two items that produced weak positive results may have come into contact with very small quantities of blood (particularly human [Chapter 7.7]), or perhaps other compounds known to produce false results (i.e., chlorophyll, plant peroxiodase, or iron compounds). Because of the latter possibility, they are not regarded to be conclusive indication of haemoglobin presence.

To test the possibility that chlorophyll might produce a false positive reaction for tools that were engaged in working of woody plants, the Hemastix test was performed on abundant residues adhering to the ventral face of the distal projection of experimental tool (E.T.) #8, which was used to scrape saskatoon bark and wood (Chapter 7; Appendix 9). The negative results obtained suggests that residues deposited on stone tools engaged in working woody plants do not produce a false positive reaction for the Hemastix test, and this potential source of error can be discounted. The Hemastix test was also performed on bone and antler shavings; negative results were also obtained for these materials.

5.2.2 Results of the Iodine Test

Thirteen (72%) of the eighteen tools tested for starch produced positive results (Table 3). A weak positive reaction was recorded if one to three stained particles (i.e., individual starch grains) were observed, a moderate reaction for four to six particles, and a strong reaction for more than six particles. Seven tools produced a weak positive result, four were associated with a moderate reaction, and two yielded a strong reaction. Saskatoon bark and wood shavings were tested with this reagent, and a strong positive reaction was observed. Bone and antler shavings were also tested; negative results were obtained.

5.2.3 Results of the Phloroglucinol-HCl Acid Test

Fourteen (78%) of the eighteen tools subjected to this test produced positive results for the presence of plant parts (Table 3). A weak positive reaction was observed if less than five tiny, translucent, organic-looking stained particles were apparent. Moderate results were recorded if more than five such tiny particles were observed. A strong positive result was recorded if the sample

contained obvious stained plant parts (i.e., wood fibres) and five or more tiny, translucent, organic-looking stained particles. Nine tools yielded a weak positive reaction, one from Kamloops (EeRb 10:6) produced a moderate positive result, and three from the Mid-Fraser River region (EeRk 4:1-51, EeRk 4: 10-1007 and EeRl 4:364) produced strong positive results.

Tools associated with moderate or strong positive reaction results bore residues that, when scraped from step- or hinge-terminated flake scars along the functional tool edges at 20 X magnification, contained visually apparent plant fibres measuring between about .1 and 2 mm long completely embedded within a sticky resinous substrate. It was concluded that these were wood fibres because: (1) most were elongate and/or cylindrical and somewhat "kinky" in form with either frayed or invasive terminating ends; (2) they were highly cohesive and relatively resistant to mechanical pressure; and (3) they were semi-translucent, and very pale yellow or white in colour. Subsequent reagent testing confirmed their identity as plant matter. Numerous tiny (less than .1 mm) plant/wood fragments were also observed.

The sticky resinous substrate deposit containing the embedded wood fibres and plant matter observed adhering within the flake scar terminations of the prehistoric specimens was identical to residues observed on the surfaces of all experimental tools used to work wood in this study (Chapters 7.6 and 7.7; Figure 66). Loy (1983) has also observed plant residues on prehistoric stone tools from northern B.C..

Obvious wood fibres were not visually detected in residues scraped from tools producing weak positive reactions. Nevertheless, once tested, between two and five tiny fragments of stained plant parts could be easily discerned at a magnification of 40 X.

The Phloroglucinol-HCl acid reagent was used to test dried saskatoon bark and wood shavings; very strong positive results were obtained. It was also used on bone and antler shavings to determine whether a false positive reaction can occur for these materials, however, both tested negative.

5.3 Summary and Discussion

The results of the residue analyses indicate that sixteen (88%) of the eighteen tested key-shaped formed unifaces bore traces of plant lignin and/or starch in residues accumulated within step- and hinge-terminated flake scars on the dorsal surface of the tool along the concave margin. Notable concentrations of plant lignin and/or starch were detected on five items, including EeRb 10:6, EeRk 4:1-51, EeRk 4:19-1007, EeRk 4:19-2119, and EeRl 4:364. Only three tools (16%) tested positive for the presence of blood (haemoglobin), although two of them produced weak reactions that can be questioned due to other factors known to produce false positive reactions. The analyses demonstrate that the main working edges of the tested sample bore residues containing plant remains.

It might be argued that plant remains could have accumulated on these tool edges post-depositionally as a result of being buried in soils containing large quantities of naturally present plant matter. However, this hypothesis is regarded to be unlikely. As mentioned above, while mechanically removing residue samples from the dorsal surface of the concave margin of several tools with a stencil knife, it was noted that dark brown residues adhering to the immediate tool surfaces had a tacky tar-like consistency typical of hardened plant resins.

Tiny wood and plant fibres and small grains of silt and sand were readily observed embedded in this resinous substrate. Most silt and sand grains were encountered within the outer portion of the deposits, whereas the plant remains were embedded throughout. This residue deposit did not resemble, in any manner, the nature of any commonly occurring chemical precipitates (e.g., calcium carbonate and ferrous compounds) found in typical soil matrices on the Plateau that might otherwise be responsible for adhering non-culturally introduced plant remains to these tool surfaces.

I conclude that the presence of wood and plant fibres, starch, and resinous deposits on the concave margin of these tools is best explained by inferring their direct involvement in plant processing activities rather than resulting from post-depositional agencies. Identical deposits were shown to accrue on surfaces and in snap- and hinge-terminated flake scars on all key-shaped formed unifaces used to strip bark and shave, scrape, and plane wood in the experimental component of this study, even after they were extensively cleaned with acetone (Chapters 7.6 and 7.7; Figure 66).

Results of the residue analysis should not be regarded as conclusive evidence for indicating the primary function of prehistoric key-shaped formed unifaces. It is viewed as an adjunct inquiry to be considered along with, and compared to, results of the design theory analysis (Chapter 3.3), microwear analysis (Chapter 6), and experimental analysis (Chapter 7) before any definitive conclusions or inferences can be drawn.