Accessing Hunter-Gatherer site structures using Fourier transform infrared spectroscopy: applications at a Taltheilei settlement in the Canadian Sub-Arctic

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The results of Fourier transform infrared spectroscopy on soils and caribou bone from a Taltheilei culture settlement in northern Canada contribute to developing micro-archaeological approaches suitable for locating and characterizing hearth and midden features on hunter-gatherer sites. A weak yet pervasive signal for montgomeryite was developed from the diagenesis of dispersed ash and caribou processing residues. Disordered calcite, carbonate hydroxylapatite, charcoal, and burned bone in two pit-house hearth deposits indicate that both wood and bone were used for fuel. Crystallinity indices and carbonate/phosphate ratios for bone indicate high intensity burning. These data, in tandem with the presence of semi-subterranean dwellings, demonstrate that this particular tundra-based encampment was occupied during cold seasons, a type of settlement behaviour previously unrecognized in the Taltheilei archaeological record. Our results confirm that Fourier transform infrared spectroscopy is an accessible, rapid, and cost effective means of discovering micro-archaeological evidence valuable for reconstructing hunter-gatherer site structures.

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1. Introduction

1.1. Objectives

The archaeological record of Taltheilei caribou hunters (ca. 600 B.C.–A.D. 1700) is gradually being uncovered in southern Nunavut and northern Manitoba, Canada (Gordon, 1975, 1996; Nash, 1970; Petch, 1992; Petch et al., 1997). Taltheilei sites in the region are generally lithic scatters, stone markers, or small sites with low artefact densities that are interpreted as ephemeral camps (Schwimmer et al., 1998). Recent surveys and excavations in southern Nunavut have discovered new evidence documenting Taltheilei land-use, specifically the extended cold season use of tundra-based pit-house settlements (Dawson et al., 2009; Hodgetts et al., 2011). These sites provide unique opportunities to document the use and organization of space at the settlement scale. Locating and characterizing hearth and refuse disposal areas, in particular, are fundamental to accurately reconstructing space at hunter-gatherer settlements (Binford, 1983; Oswald, 1984). Centring on the micro-archaeological record at the Ikirahak settlement (JjKs-7) in southern Nunavut, this research uses Fourier transform infrared spectroscopic (FTIR) analyses of soils and caribou bone to characterize dwelling hearth features and to locate outdoor hearths and bone middens. We aim to determine the use of space at the site and clarify its season of occupation, which will contribute both to addressing issues with our understanding of Taltheilei settlement—subsistence patterns and to developing micro-archaeological approaches suitable for defining hunter-gatherer site structures.

Frequencies, sizes, and locations of hearth and midden features are useful for reconstructing dynamics in site functions, seasons of occupation, mobility patterns, and socio-economics. The types of materials used for fuel can provide evidence for seasonality, while multiple overlapping hearths act as a record of reoccupation (Bamforth et al., 2005; Schiegl et al., 2003). Similarly, numerous large middens are common at long-term camp sites and at sites with long histories of reoccupation. Shorter term camps tend to develop a light layer of homogeneously dispersed refuse (Kent, 1999). Smaller middens situated adjacent to dwellings typically represent household work and refuse disposal, while larger middens in central and peripheral areas can relate to communal activities (Beck and Hill, 2004; Oetelaar, 1993).
Hearths and middens are often faintly expressed or even invisible in the archaeological record, and their locations may only be defined through micro-archaeological techniques such as soil/sediment chemistry (Middleton and Price, 1996). Fourier transform infrared spectroscopy of soils, sediments, and reference materials is an accessible, rapid, and cost-effective means of locating fading hearth and refuse disposal features on hunter-gatherer sites. Here we use FTIR to define the impact of burning and bone decay on the mineral composition of soils from Ikirahak’s dwelling hearths and outdoor spaces and to determine the temperatures bones excavated from hearths were exposed to during burning. The influence of burning on soils is investigated using traces of calcite, carbonate hydroxylapatite, and hematite, while the effects of bone decomposition are explored using the presence of authigenic phosphates (Karkanas et al., 2007; Weiner et al., 1993). Bone burning is evaluated using the crystallinity index, the carbonate/phosphate ratio, and changes in organic matter, collagen, and phosphate peak characteristics (Squires et al., 2011; Thompson et al., 2009). Our research is among the first using FTIR to characterize materials from an archaeological site in the Canadian Arctic (e.g., Helwig et al., 2008) and it provides preliminary evidence for the formation of authigenic montgomeryite on an open-air archaeological site.

### 1.2. Taltheilei caribou hunters

The Taltheilei archaeological culture was defined through excavations at several sites around the Taltheilei Narrows of Great Slave Lake (Nash, 1970). Its roots originate in the forests of the Peace River Valley, northern British Columbia and Alberta (Gordon, 1975). Between 700 and 600 B.C. pioneers moved northeast into the Beverly Caribou Range. Radiocarbon dates from forest sites on the Dubawnt River demonstrate that the earliest Taltheilei groups rapidly populated the area and adapted to hunting caribou around 575 B.C. (Gordon, 1996) (Fig. 1). Gordon (1996) argues that throughout their roughly 2300 year occupation of the region, they spent spring and fall following caribou to and from the tundra respectively. During midsummer, they lived on the tundra in small, highly mobile groups. Winters were passed in the forest in larger, more sedentary groups.

Over 1000 Taltheilei sites (ca. 600 B.C.—A.D. 1700) have been discovered throughout Beverly Caribou Range’s tundra and boreal forest landscapes (Gordon, 1996). Far less research has been undertaken in the Kaminuriak Caribou Range, though recent archaeological surveys around Maguse Lake identified several pit-house sites (Dawson et al., 2009; Hodgetts et al., 2011) (Fig. 1). These are among the first identified Taltheilei sites of their kind. The Ikirahak site (JJKs-7), the focus of this paper, is a tundra-based pit-house settlement located on the southwestern shoreline of a small island that sits in the Narrows of Maguse Lake near a large caribou crossing. Positioned on a gently sloping terrace approximately 10 m above the lake shore, the site consists of 10 pit-house depressions and several potential storage features (Fig. 2). Diagnostic points and hide abraders, living floors, and AMS dates on caribou bone indicate that Taltheilei people occupied the settlement several times between approximately A.D. 500 and 650.

An investment into building pit-houses supports the argument that this tundra-based settlement was occupied for a longer period of time during a cold season (Binford, 1990; Kelly et al., 2005; LeMouel and LeMouel, 2001; McGuire and Schiffer, 1983). This differs significantly from the settlement pattern documented in the Beverly Caribou Range, where the tundra was occupied by small mobile groups during the summer. The houses are each approximately 4.5 m in diameter, and they were constructed by excavating a roughly 50 cm deep circular pit and banking the sediments around the edge. Boulders were placed around the perimeter of the sediment berms to secure hide superstructures. The rocks were pushed into the centres of the dwellings upon abandonment. A group of caribou hunters occupying the site for an extended period would produce a substantial amount of refuse, yet there are no discernible bone middens at Ikirahak. Bone may have decomposed and reprecipitated in soils as authigenic phosphates and/or it was...
used as fuel, both of which are evaluated below using FTIR. Analyses of burned bone will also help clarify whether the site was occupied during a cold season.

2. Locating hearth and bone midden features using FTIR

2.1. FTIR

Fourier transform infrared spectroscopy measures the vibrational behaviours of chemical bonds (Bhargava and Levin, 2005). Infrared (IR) light passing through a molecule causes its bonds to vibrate in characteristic ways, such as asymmetric stretching. Vibrations are recorded as frequencies that are Fourier transformed into wavenumbers (the inverse of wavelength) (Smith, 2011). Specific types of bonds absorb characteristic amounts of infrared radiation at diagnostic wavenumbers between 250 and 4000 cm$^{-1}$ (Harwood and Claridge, 1997). A characteristic carbon–oxygen bond in carbonate, for instance, exhibits an asymmetric stretch between 1415 and 1420 cm$^{-1}$ (Tatzber et al., 2007).

The strengths and applications of FTIR are far-reaching and include identifying functional groups of molecules, defining atomic structures, and characterizing unknown materials. Straightforward sample preparation and instrument operation procedures make FTIR analyses of systematically collected soil/sediment samples an accessible and rapid means of characterizing and locating a variety of feature types. Three-dimensional spatial analyses of mineral assemblages are particularly valuable in reconstructions of both site formation processes and the structured use of space (Weiner et al., 2002). Instruments measure a range of wavenumbers simultaneously and they rapidly take multiple measurements of samples, providing averaged spectra with optimal signal-to-noise ratios (Bhargava and Levin, 2005). In addition, instruments produce accurate and replicable results in laboratory and field contexts (Shahack-Gross et al., 2005), they simultaneously measure organic and inorganic compounds (Thompson et al., 2009), and they identify atomically disordered minerals such as calcite derived from wood ash, authigenic phosphates from decomposing ash and bone, and carbonate hydroxyapatite in bone (Karkanas et al., 2000; Ikirahak (JjkS-7) site plan.
Weiner, 2010). Drawbacks include intra-sample variation owing to tiny aliquot sizes, shifting peak maxima owing to variation in grinding, and overlapping absorbance peaks in mixtures such as soils (Smith, 2011).

2.2. Hearth features

The clay, iron, and calcite components of soils and sediments, defined using IR spectra, are useful for distinguishing the locations of primary hearth features and secondary ash dumps when visible material evidence is faded or erased. Intense heat causes disorder in the atomic structures of clay minerals in adjacent soils and sediments. Montmorillonite, for instance, undergoes several diagnostic structural changes between 400 and 1100 °C. Disorder in the mineral, indicated by shifting peaks, the disappearance of peaks, and the appearance of sharp peaks, is related to the temperature of the heat source, distance from the heat source, and the duration of exposure (Berna et al., 2007). Rubification owing to high concentrations of hematite also helps distinguish primary hearth contexts. Acidic and reducing conditions caused by burning increase the solubility of iron in the deposit. Reduced iron tends to dissolve and re-oxidize into the insoluble iron mineral hematite, which both redoxes the deposit and enhances its magnetic signal (Aspinall et al., 2008; Karkanas et al., 2002). Heat altered hematite has several sharp IR absorbance bands between 650 and 400 cm⁻¹ (Rendon and Serna, 1981).

A more frequently applied approach to defining hearths using FTIR focuses on disordered calcite in soils and sediments (Albert et al., 2003; Goldberg et al., 2012; Karkanas et al., 2002; Schiefelbein et al., 2003; Weiner et al., 1995, 1998, 2002). Burning wood at temperatures between 400 and 500 °C transforms its calcium oxalate component into calcite through the expulsion of carbon monoxide (Huaqing, 1989; Regev et al., 2010). Calcite absorbs at 1420 cm⁻¹ (v3, asymmetric stretch), 874 cm⁻¹ (v2, out-of-plane bending), and 713 cm⁻¹ (v4, in-plane bending) (Chu et al., 2008). The ratio of the v2 and v4 peaks distinguishes the mineral’s level of atomic disorder. Ash calcite is disordered, while the geogenic polymorph is much more crystalline (Regev et al., 2010).

Pyrogenic calcite, however, may not survive site forming processes (Karkanas et al., 2007). It is relatively soluble and unstable, specifically in the acidic, organically enriched conditions caused by burning. In acidic deposits rich in phosphates, amorphous calcite can be transformed into the more stable authigenic carbonate hydroxylapatite (Weiner et al., 1993). The formation of such authigenic phosphates in soils and sediments is primarily linked to acidity, phosphate levels in the system’s water, mineral stability, and available carbonates (Karkanas et al., 2000; Nriagu, 1976). Decomposing human and animal wastes, vegetation, bone, and many other types of organic matter add significant amounts of phosphate to soils and sediments, which in turn causes a reduction in the pH of the deposit (Goldberg and Nathan, 1975). These conditions contribute to the diagenesis and reprecipitation of carbonates, including amorphous ash calcite and carbonate hydroxylapatite from decaying bones, into authigenic phosphates (Karkanas et al., 2002).

2.3. Bone as a fuel source

Hearth features containing few wood charcoal fragments and minor chemical traces of calcite, but abundant calcined bone fragments and strong signatures for primary carbonate hydroxylapatite, indicate that both bone and wood were burned, yet bone was the principal fuel source (Schiefelbein et al., 2003). Stages of bone burning and natural decomposition can be identified using IR spectra to define the crystallinity index (CI, also called the splitting factor), the carbonate/phosphate ratio (C/P), and organic matter, collagen, and phosphate peak changes (Thompson et al., 2009).

Several recent studies detail the strengths and weaknesses of the CI for defining the characteristics of burned bone from both archaeological and experimental contexts (Lebon et al., 2010; Squires et al., 2011; Thompson et al., 2009, 2010). The degree of splitting between the peaks of the carbonate hydroxylapatite doublet at 605 and 565 cm⁻¹ corresponds to the organization of bone tissue’s atomic structure (Nagy et al., 2008; Weiner and Bar-Yosef, 1990). As natural decomposition or burning progresses, the atomic structure of carbonate hydroxylapatite becomes more organized (Stiner et al., 1995; Weiner and Price, 1986). Mineral crystals become larger, more crystalline, and more chemically stable (Shipman et al., 1984). Sharper absorption peaks at 605 and 565 cm⁻¹ indicate an ordered crystalline structure (Thompson et al., 2009). Changes in peak sharpness are a function of atomic alterations caused by burning, but they are also caused by the slower processes of chemical diagenesis (Stiner et al., 2001). As such, the crystallinity index alone may not be an accurate indicator of bone burning (Lebon et al., 2010; Trueman et al., 2008). Here, it is supplemented with the C/P ratio and changes in organic matter, collagen, and phosphate peak characteristics (Weiner, 2010).

The C/P ratio provides an estimate of the amount of carbonate in a sample (Thompson et al., 2009). It decreases as the carbonate fraction decreases (Squires et al., 2011). The phosphate component used in the ratio absorbs between 1035 and 1048 cm⁻¹ while the carbonate component absorbs between 1415 and 1420 cm⁻¹. Both highly burned and decomposed bone contain close to no carbonate, indicated by an absence of peaks at 872, 1420, and 1456 cm⁻¹ (Stiner et al., 1995). Bones burned at high temperatures, typically exceeding 700 °C, also have sharper, narrower phosphate peaks (Thompson et al., 2009). Moreover, the phosphate peak shifts from 1035 cm⁻¹ in unaltered bone to 1048 cm⁻¹ in highly burned bone (Weiner, 2010). The organic matter doublet at 2924/2853 cm⁻¹ and collagen peaks at 1750–1550 cm⁻¹ and 1455 cm⁻¹, representing amide I and carbonyl functional groups respectively, are drastically reduced in highly mineralized/burned bone. Collagen peaks are primarily absent in samples that have been heated above 700 °C (Thompson et al., 2009). The appearance of sharp peaks at roughly 632 and 1090 cm⁻¹ represent the formation of crystalline phosphate and apatite phases, making them some of the best indicators of calcined bone (Weiner, 2010).

2.4. Bone diagenesis after burial

Infra-red spectra of soils and sediments can help pin-point locations where bone was deposited and subsequently decomposed, or where ash calcite has undergone significant diagenesis and reprecipitation (Berna et al., 2004; Weiner et al., 1993). Bone collagen decomposes in alkaline soil/sediment environments, leaving a carbonate hydroxylapatite mineral ghost with low porosity (Collins et al., 2002; Nielsen-Marsh and Hedges, 1999). The carbonate hydroxylapatite component decomposes in acidic, aerobic deposits (Cronyn, 1990; Hedges, 2002). Under these circumstances, bone gradually dissolves and its carbonate mineral products can react with available phosphates to form relatively insoluble authigenic phosphates (Karkanas et al., 1999; Schiefelbein et al., 1996).

A reaction cascade for authigenic phosphates in archaeological cave sediments has been defined and used to establish where bones were deposited, completely decayed, and reprecipitated as new mineral formations, to distinguish primary from secondary deposits, and to distinguish episodes of occupation and abandonment (Karkanas et al., 2002; Schiefelbein et al., 1996; Weiner et al., 1993). Crandallite forms in acidic deposits (pH ~ 4–6) with high aluminum and calcium yet low phosphate levels, while montgomeryite forms in...
acids (pH $\sim 4–6$) with high phosphate, calcium, aluminum, and magnesium levels (Karkanas et al., 2000; Nriagu, 1976; Weiner, 2010). Variscite and tarañakite can form after prolonged alteration of montgomeryite or crandallite in highly acidic deposits (pH < 4) with elevated phosphate concentrations (Goldberg and Nathan, 1975; Karkanas et al., 2002). Tarañakite can also form directly from carbonated hydroxyapatite in neutral pH environments (Karkanas et al., 2000). Authigenic phosphates reprecipitated from calcite or carbonate hydroxyapatite can be atomically disordered. Peaks for the disordered montgomeryite polymorph forming from these minerals characteristically appear at 1070, 1058, and 595 cm$^{-1}$, and also at approximately 3413, 1643, 1036, 590, and 562 cm$^{-1}$ (Weiner et al., 2002; Weiner, 2010).

3. Materials and methods

3.1. Sampling and processing

One hundred forty-five soil samples were collected from the settlement on a lattice-grid using an Oakfield soil corer. A 3 m sample interval was chosen to balance the size of the sampling universe ($\sim$5 km$^2$), the potential sizes of areas influenced by outdoor hearths and bone middens, and time in the field (Fig. 2). Some gaps in our grid exist because samples could not be taken from every point. Some areas were too rocky, had poor soil formation, were disturbed by excessive cryoturbation, or overgrown with brush. During excavations, samples were collected from the hearths of Houses 3 and 8 and from a potential hearth in House 2. Addressing intra-sample variation, we first homogenized our large bulk samples using a porcelain pestle and mortar. We separated the fine fractions using a 2 mm sieve and then micro-chute split (riffling) them to reduce the sample size. Aliquots were taken from the reduced samples and oven dried at 120 °C for 24 h to remove gravimetric water.

Organic matter contents were determined using the loss-on-ignition method and particle size analyses were conducted using a Malvern laser diffraction system. The pH and Eh were recorded for each sample in a 1:2 mixture of soil and distilled water using potentiometric soil meters. Element concentrations were measured as the percent weight of their oxides using x-ray fluorescence (see Butler, 2011).

Archaeological caribou bone samples (Rangifer tarandus groenlandicus) were collected during the excavations of Houses 2, 3, and 8 (floor N = 16; hearth N = 6). Fresh (N = 4), burned (N = 3), and buried (N = 1) comparative samples were collected from two recent caribou processing camps near the hamlet of Arviat. To reduce issues caused by intra-sample variation, we selected relatively large samples for crushing. Samples were crushed using a Carver Laboratory press. We homogenized the particles using a porcelain pestle and mortar and then separated a very fine fraction using a 0.15 mm sieve. This fraction was micro-chute split and an aliquot was taken from the reduced sample. Samples were also characterized using preservation categories described by Haynes et al. (2002).

3.2. FTIR analyses

Absorption spectra were collected using the potassium bromide (KBr) disk method (Surovell and Stiner, 2001). Approximately 300 mg of KBr powder was gently ground and homogenized with 2–4 mg of sample. Although grinding was facilitated manually using a porcelain pestle and mortar, we aimed to reduce the effects of shifting peak maxima owing to variation in grinding by practicing and duplicating the appropriate grinding force, pattern, and timing. Roughly half the mixture was evenly spread inside a KBr die and pressed at 13,000 lbs on a Carver Press under a vacuum for 60 s.

Spectra were collected using a Nicolet Nexus 470 operated by OMNIC™ 6.2 software. A background spectrum was collected every 100 min and a nitrogen gas flow was used to eliminate interference from atmospheric water and carbon dioxide. Spectra were derived from an average of 64 scans at 4 cm$^{-1}$ resolution, they were linear baseline corrected, and they were smoothed using 13 points.

3.3. Hearth and midden features

Traces of amorphous calcite, primary carbonate hydroxyapatite, and hematite were used to search for outdoor hearth areas. Laser diffraction particle size analyses did not detect any clay in the samples, excluding the use of clay minerals to identify hearth locations. Calcite was defined in the soil samples using its characteristic peaks at 1420 cm$^{-1}$ (v3), 874 cm$^{-1}$ (v2), and 713 cm$^{-1}$ (v4). Following Chu et al. (2008), we used the ratio of the v2 and v4 peak heights to determine whether any discovered calcite is wood ash or geological. Measuring baselines were drawn from the lowest points of the valleys bookending the peaks. Geological calcite has values around 3.0, while values for ash calcite are around 4.0. The presence of heat altered hematite was assessed using sharp peaks at 650, 555, 525, 470, 440, and 400 cm$^{-1}$ (Rendon and Seria, 1981).

We explored the site for diagenetically transformed bone middens using authigenic phosphates. To identify these minerals, we compared our spectra with literature data (Karkanas et al., 2002; Schiegl et al., 1996; Weiner et al., 2002; Weiner, 2010) and with archived reference spectra (KCAS, n.d.) for montgomeryite, crandallite, variscite, and tarañakite. Areas that have no evidence of bone fragments, carbonate hydroxyapatite, or authigenic phosphates are considered free of alteration caused by the deposition, diagenesis, and reprecipitation of bone mineral. An absence of calcite and carbonate hydroxyapatite along with the presence of disordered montgomeryite will indicate the diagenesis and transformation of phosphates and carbonates in an acidic environment. On archaeological sites, refuse from human activities, specifically bone and ash particulates, provides abundant phosphates, carbonates, and magnesium that may contribute to the formation of authigenic phosphates (Goldberg and Nathan, 1975). The presence of authigenic montgomeryite in an archaeological context is a dependable indicator that bone and/or ash were deposited but did not preserve (Karkanas et al., 2007; Weiner et al., 1993).

3.4. Bone crystallinity

Crystallinity indices were calculated by dividing the sum of the 605 and 565 cm$^{-1}$ peak heights by the height of the valley separating them (Weiner and Bar-Yosef, 1990). Large values indicate significant decomposition and structural ordering of the bone apatite (Lebon et al., 2010; Munro et al., 2007). Values for modern unaltered bone typically lie between 2.5 and 3.5. Values for moderately decomposed or burned bone range between approximately 3.6 and 4.5, while those for highly decomposed or burned bone range between 5 and 7 (Thompson et al., 2009).

Carbonate/phosphate ratios were calculated by dividing the height of the carbonate peak at 1420 cm$^{-1}$ with the height of the phosphate peak at 1035 cm$^{-1}$ (Squires et al., 2011). Unaltered bone has a ratio between 0.31 and 0.65. Decreasing values are a function of natural diagenesis, but they are exacerbated by burning. Values as low as 0.04 indicate burning at temperatures surpassing 700 °C (Thompson et al., 2009). Supplementing the CI and C/P ratio, degrees of burning/mineralization were classified using changes in organic matter, collagen, and phosphate peaks. We use the decomposition of the organic matter doublet at 2924/2853 cm$^{-1}$ and collagen peaks at 1750–1550 cm$^{-1}$ and 1455 cm$^{-1}$, along with sharper, narrower, left-shifted phosphate peaks to identify highly
mineralized/burned bone. Additionally, the formation of sharp peaks on the carbonate hydroxylapatite doublet at 632 cm\(^{-1}\) and on the phosphate peak at 1090 cm\(^{-1}\) will indicate the formation of crystalline mineral phases (Weiner, 2010).

Descriptive statistics and analysis of variance (ANOVA) undertaken using S-Plus 8.0 and SPSS 16.0 were used to distinguish groups of unaltered, buried, and burned bone. The analysis tests the null hypothesis that there is no difference in CI and C/P values between the groups using the F-test to compare the variances of means within groups and between groups. To satisfy statistical assumptions and avoid type I statistical error, quantile-quantile plots were used to assess whether the sample population is normally distributed and Levene’s score was used to test for homogeneity of variances.

4. Results

4.1. Soil analyses

Soils at the site are organically enriched cryogenic silt loams. Loss-on-ignition organic matter content ranges from 16% to 83% (\(\bar{X} = 51\%\)). Laser diffraction particle size analyses indicate the soils contain an average of 42% sand and 58% silt. They are also acidic and aerobic, having pH readings between 3.76 and 5.00 (\(\bar{X} = 4.22\)) and oxidizing redox potentials between 408.30 mv and 519.50 mv (\(\bar{X} = 467.81\) mv) (Table 1). Cryoturbation is indicated by large surface features such as ice wedges and hummocks and micro-morphologically by ice wedging and planar voids in soil thin sections (McNamee et al., 2009). Charcoal is present in the House 3 and 8 hearth samples and in several cores from across the site. Burned bone fragments are present in the House 3 and 8 hearth samples.

All of the collected spectra have phosphate and carbonate components. Calcite and carbonate hydroxylapatite are present in the hearth soils of Houses 3 and 8. Calcite is disordered, with v2/v4 ratios of 4.5 for House 3 and 4.0 for House 8. Calcite, carbonate hydroxylapatite, and hematite are not present in the soils from across the site. Montgomeryite is near ubiquitous across the site, hydroxylapatite, and hematite are not present in the soils from across the site. Montgomeryite is near ubiquitous across the site, hydroxylapatite, and hematite are not present in the soils from across the site.

4.2. Bone analyses

Median, minimum, and maximum CI and C/P values are primarily distinct for the three groups, but their ranges have some overlap (Fig. 5; Table 2). Means for each group are also somewhat different but there are some issues with large standard deviations, which could be reduced by using a larger sample size. Crystallinity index means for unaltered, buried, and burned bones were 2.6, 3.2, and 4.2 respectively. The standard deviation of the unaltered bone (\(\sigma = 0.08\)) is much lower than those for the buried (\(\sigma = 0.60\)) and burned (\(\sigma = 0.88\)) bones. The highest value for CI, 5.5, is from a burned bone retrieved from the hearth in House 3. The lowest value for C/P, 0.06, is from the same sample. The C/P means are 0.62, 0.19, and 0.13 for unaltered, buried, and burned groups. Standard deviations are somewhat large, valuing 0.26 and 0.12 for unaltered and burned bone respectively. Burned bone was much lower at 0.06.

Analysis of variance results, supporting the descriptive statistics, highlight statistically significant differences in the CI and C/P means between unaltered, buried, and burned bone groups. Quantile-quantile plots display normal distributions for both CI and C/P and values for Levene’s test (\(p \leq 0.05\)) indicate homogeneous variances, both contributing to the robusticity of the analysis against type I error. F-values for CI and C/P, 13.04 and 22.43 respectively, are statistically significant and they far-exceed the critical F-value (\(F = 5.49; df = 2, 27; p < 0.01\)), indicating the groups are statistically distinct. Tukey post-hoc tests for CI confirm that unaltered and burned bone have the greatest differences. Buried and burned groups are also significantly different. Unaltered and buried groups have some overlap. Means for the C/P ratio diverge considerably between the unaltered and buried groups and between the unaltered and burned groups. Buried and burned groups have some overlap caused by their standard deviations. Based on the ANOVA and descriptive statistics, the CI distinguishes chemically decomposed bone (burned) from burned bone better than the C/P ratio in our dataset (Fig. 5).

We also documented clear changes in organic matter, collagen, and phosphate peaks (Fig. 4). The organic matter doublet is very high in fresh bone, yet significantly reduced in the burned/decomposed samples. Collagen peaks are extensively reduced in height and width during the sequence of decomposition. Samples with high CI and low C/P values have sharper phosphate peaks that are shifted slightly from the 1030 s to 1040 s cm\(^{-1}\). Three highly resolved, sharp peaks also develop throughout decompositional process, and they distinguish highly mineralized/burned bone samples. Sharp peaks appear on the left side of the phosphate peak at 962 cm\(^{-1}\) and on the right side of the phosphate peak at 1090 cm\(^{-1}\), representing the formation of a highly crystalline phosphate phase. Similarly, a highly resolved peak forms on the left side of the carbonated hydroxylapatite doublet at 632 cm\(^{-1}\), which again relates to the formation of a crystalline phase.

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<th>OM</th>
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<th>Silt</th>
<th>P(^a)</th>
<th>Ca</th>
<th>Al</th>
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<td>46.4</td>
<td>0.37</td>
<td>1.92</td>
<td>2.92</td>
<td>0.90</td>
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<tr>
<td>91</td>
<td>Light brown, silt loam</td>
<td>4.24</td>
<td>461.3</td>
<td>24%</td>
<td>27.1</td>
<td>72.9</td>
<td>0.50</td>
<td>2.00</td>
<td>3.59</td>
<td>0.69</td>
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<tr>
<td>101</td>
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<td>3.90</td>
<td>480.2</td>
<td>34%</td>
<td>53.3</td>
<td>46.7</td>
<td>0.90</td>
<td>2.80</td>
<td>8.50</td>
<td>2.76</td>
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<tr>
<td>120</td>
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<td>3.84</td>
<td>484.3</td>
<td>78%</td>
<td>59.3</td>
<td>40.7</td>
<td>0.64</td>
<td>7.93</td>
<td>0.92</td>
<td>0.21</td>
</tr>
<tr>
<td>135</td>
<td>Dark brown, silt loam</td>
<td>4.32</td>
<td>467.6</td>
<td>52%</td>
<td>9.9</td>
<td>90.1</td>
<td>0.44</td>
<td>4.83</td>
<td>0.96</td>
<td>0.32</td>
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<tr>
<td>147</td>
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<td>4.22</td>
<td>467.8</td>
<td>30%</td>
<td>20.2</td>
<td>79.8</td>
<td>0.80</td>
<td>6.77</td>
<td>2.28</td>
<td>1.05</td>
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<tr>
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<td>4.56</td>
<td>478.3</td>
<td>45%</td>
<td>40.5</td>
<td>59.5</td>
<td>0.36</td>
<td>5.51</td>
<td>0.96</td>
<td>0.23</td>
</tr>
<tr>
<td>Off site</td>
<td>Dark brown, silt loam</td>
<td>4.47</td>
<td>464.7</td>
<td>37%</td>
<td>25.1</td>
<td>74.9</td>
<td>0.42</td>
<td>1.97</td>
<td>2.09</td>
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</table>

\(\text{a Elements measured as % wt of their oxides.}\)
5. Discussion

5.1. Accessing hunter-gatherer site structures using FTIR

Fourier transform infrared spectroscopy is an accessible, rapid, and cost-effective means of collecting micro-archaeological evidence useful for uncovering the site structures of hunter-gatherer settlements. Instruments are widely available and sample processing and analyses are comparatively fast and straightforward. We processed and analysed on average 7 samples an hour and we logged 25 h, making our project both efficient and inexpensive. Considering the rapidity and low cost, soil surveys using FTIR assist the identification of areas on sites for further, more advanced and costly analyses such as inductively coupled plasma mass-spectroscopy and gas chromatography.

Fourier transform infrared spectroscopic analyses contribute to identifying the characteristics and locations of features on archaeological sites that may otherwise remain invisible. Hunter-gatherers engaged in many types of spatially organized work that did not leave behind common types of material evidence. Many
tasks did, however, introduce chemical residues into underlying soils. Facilities that may remain invisible to routine investigation yet leave chemical footprints include storage areas, hide processing areas, meat drying areas, hearth and food preparation areas, refuse disposal areas, bedded areas, and houses built from snow and ice (Butler, 2011; Knudson and Frink, 2010; Middleton and Price, 1996). These footprints, in many cases, are required to accurately reconstruct the range of activities represented at settlements. Infrared spectra of soils, sediments, and reference materials provide data useful for distinguishing the footprints of various types of high and low diversity work spaces. As mentioned above, however, soils are complex mixtures of compounds, many of which absorb in similar areas of the IR spectrum. Overlapping absorbance peaks are sometimes difficult to identify, and they can, along with poor quality spectra, lead to the misidentification of the compounds present in a complex mixture like soil.

The chemical record at Ikirahak is clear in our IR spectra, which owes to the high quality of our KBr discs. Disks were primarily clear and they produced spectra having absorbencies below one, yet not below zero, indicating the proper ratio of KBr and sample was used.

![Fig. 4. Representative FTIR spectra for bone.](image-url)
It is not uncommon to use a low number of scans to increase productivity and minimize costs, and our spectra show that some of these spectra can be used to achieve high quality results with a reduced number of scans. This is particularly true when using a baseline correction method, which can help to reduce noise and improve the quality of the resulting spectra.

Diagnostic peaks for calcite and those used to classify degrees of bone decomposition/burning are well documented in the literature, and they are clearly resolved in our spectra (Chu et al., 2008; Squires et al., 2011; Weiner and Bar-Yosef, 1990). We are also confident that absorptions in our samples at 3413, 2348, 1643, 1384, 1080–1070, 1058, 1035, 908, 797, 590–595, 562, 455, 423 cm\(^{-1}\) represent montgomeryite. Our spectra share many similarities with those reported by Weiner et al. (2002) and Weiner (2010). Peaks at 1080–1070, 1058, and 595 cm\(^{-1}\) are particularly diagnostic of montgomeryite (Weiner et al., 2002). Defining the mineral assemblage of the soils also helps clarify any potential ambiguity. Silicates are common in soils, yet do not absorb in the same locations as montgomeryite. Iron oxides, also very common in soils, cause broad, strong absorptions below 700 cm\(^{-1}\). These peaks are not dominating this region our spectra. Clay minerals absorb in similar wavenumber ranges as montgomeryite, though, in our study, it is not possible that absorptions from clay minerals are leading to the misidentification of montgomeryite because particle size analyses show there has been no clay formation, owing to reduced chemical weathering caused by cold temperatures. Humic acid is common in our spectra, and there is some potential overlap with montgomeryite absorptions. Despite this, peaks for both compounds are generally distinguishable. In Sample 8, for example, the peak for humic acid at 475 cm\(^{-1}\) appears as a shoulder on the left side of the 455 cm\(^{-1}\) montgomeryite peak, while the 535 cm\(^{-1}\) humic acid peak appears as a shoulder on the right side of the 562 cm\(^{-1}\) montgomeryite peak (Fig. 3). X-ray fluorescence also confirms the soils contain abundant aluminum, phosphorus, calcium, and magnesium, which are necessary for the formation of montgomeryite.

5.2. The structure of cold season stays at Ikirahak

Based on the results presented above, we propose that spatially constrained bone middens were never formed at the site, that a substantial amount of bone refuse was used as fuel, that hearth fires were hot enough to calcine bone, and that hearths were restricted to dwelling interiors. The acidity, mineral components, and element contents of the soils provide an environment suitable for the formation of authigenic phosphates, particularly montgomeryite.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Context</th>
<th>Description</th>
<th>CI</th>
<th>C/P</th>
</tr>
</thead>
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<td>Arviat; Recent Processing Camp 2 Surface</td>
<td>Fresh; robust edges; shiny, smooth, greasy surface</td>
<td>2.6</td>
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<td>0.53</td>
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<td>2.5</td>
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</tr>
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</tr>
<tr>
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</tr>
<tr>
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<td>13</td>
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<td>0.16</td>
</tr>
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<td>14</td>
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<td>0.14</td>
</tr>
<tr>
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<td>17</td>
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<tr>
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<td>4.0</td>
<td>0.14</td>
</tr>
<tr>
<td>22</td>
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<td>4.4</td>
<td>0.12</td>
</tr>
<tr>
<td>23</td>
<td>Ikirahak; House 8; Floor; Buried</td>
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<td>4.8</td>
<td>0.16</td>
</tr>
<tr>
<td>24</td>
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<td>Burned; black; fragile edges; smooth, dull surface</td>
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<td>0.28</td>
</tr>
<tr>
<td>25</td>
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<td>0.15</td>
</tr>
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<td>26</td>
<td>Ikirahak; House 8; Hearth; Buried</td>
<td>Burned; black; fragile edges; rough, dull surface</td>
<td>4.0</td>
<td>0.14</td>
</tr>
<tr>
<td>27</td>
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<td>0.07</td>
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<td>5.1</td>
<td>0.10</td>
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<tr>
<td>30</td>
<td>Ikirahak; House 3; Hearth; Buried</td>
<td>Burned; white/grey; fragmented; friable, crumbling edges</td>
<td>4.2</td>
<td>0.10</td>
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</table>
Montgomeryite is widespread across the site, making it unlikely that it formed as a result of decomposing bone discarded in spatially discrete midden areas. There are no spatially patterned traces of bone fragments or carbonate hydroxyapatite to support spatially organized bone deposition. Moreover, the soils are frozen for most of the year, and they are moderately acidic and oxidizing, meaning large fragments of buried bone would survive in this context (Table 1). It is also unlikely that montgomeryite formed naturally through geological processes. Montgomeryite can form in calcareous, clay rich soils associated with phosphatic sedimentary rocks (Nriagu, 1976); yet, the deposit at Ikirahak would not facilitate the development of montgomeryite in this way because it lacks geogenic carbonate (no visible nodules or reaction with dilute HCL), it lacks clay, and local parent geology primarily consists of granitic tills and mafic igneous bedrock (Hodgetts et al., 2011).

An alternative explanation is that montgomeryite is a product of the diagenesis of dispersed ash and caribou processing residues caused by the organically enriched, acidic, and aerobic soils across the site (Table 1; Fig. 3). Ash, flesh, bone, skin, and hair particulates, bodily fluids, and other agents contributed the carbonate phosphates, phosphates, and magnesium necessary to form a weak yet pervasive signal for montgomeryite. Traces of charcoal in several core samples support that particulates were dispersed and deposited across the site. Moreover, X-ray fluorescence results show generally enhanced concentrations of phosphorus, aluminum, magnesium, and calcium across the site. As mentioned, these elements are required for the formation of montgomeryite. More importantly, enrichments in their concentrations have been widely documented in association with the deposition of ash, charcoal, and liquid and particulate residues from animal processing (Knudson and Frink, 2010; Middleton and Price, 1996). Ash, charcoal, and decomposing organic matter from caribou hide and meat processing lowered the pH of the deposit and provided an abundance of mobile phosphorus, calcium, and magnesium that interacted with geogenic aluminum to start a reaction cascade culminating in the formation of montgomeryite.

Montgomeryite is absent in the dwelling samples, yet unstable disordered calcite is present, demonstrating their soils are more chemically stable and less altered than those from the rest of the site. Soils inside the dwellings contain more organic matter and are chemically stable and less altered than those from the rest of the site. Phosphorus, calcium, and magnesium that interacted with geochemical organic matter from caribou hide and meat processing lowered the pH of the deposit and provided an abundance of mobile phosphorus, calcium, and magnesium that interacted with geogenic aluminum to start a reaction cascade culminating in the formation of montgomeryite.

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Montgomeryite is absent in the dwelling samples, yet unstable disordered calcite is present, demonstrating their soils are more chemically stable and less altered than those from the rest of the site. Soils inside the dwellings contain more organic matter and water, they are deeper, and they have good vegetation cover, making them colder for longer periods, interrupting mineral diagenesis, and protecting them from the weathering processes affecting shallower deposits throughout the site (Karkanas et al., 1999). Caribou processing residues and ash calcite dispersed over the site experienced a greater degree of weathering and diagenesis than the amorphous calcite and bone derived carbonate hydroxyapatite in the colder, unexposed hearth deposits. Although evidence for authigenic phosphates in various soils is available, additional research using experimental deposits, micromorphology, electron microprobe, and lysimetry will clarify the processes involved with their formation and behaviour on open-air archaeological sites. Distinguishing those formed from ash and bone is particularly important.

Several formation processes would have contributed to removing bone refuse from the site. Soil forming processes and sedimentation rates are rather slow at Ikirahak, so it is unlikely that large bone fragments were buried. Furthermore, dogs were commonly kept by historic period Chipewyan people (Hearne, 1958). If Talthielei people, the ancestors of the Chipewyan, also kept dogs, they would have fed them food refuse. Similarly, after abandonment some remains would have been removed by scavenging animals. People may have also dumped refuse in the lake, or near the lake edge, where it was subsequently removed by ice rafting.

In addition, our results confirm the use of bone, and wood, to fuel dwelling hearths. People likely built hearths using drift wood and dwarf willow, adding bone once a sufficiently hot core was established. Two of the three tested hearths (Houses 3 and 8) contain chemical traces of both disordered calcite and carbonate hydroxyapatite, as well as fragments of wood charcoal and burned bone (Fig. 3). Calcite in the hearths, based on the v2/v4 ratios, is pyrogenic. Carbonate hydroxyapatite in hearth soils was derived from burned bone fragments and powder, not from the dissolution and reprecipitation of ash calcite (Schiegl et al., 2003). Ash calcite is stable in the dwelling deposits, indicating it has not undergone transformation into carbonate hydroxyapatite. The carbonate hydroxyapatite identified in the hearth samples was derived from the calcined bone powder produced when bone is burned at high temperatures (Stiner et al., 1995). Calcined bone fragments were further powdered and incorporated into the soil matrix via cryoturbation. The absence of material and chemical evidence for a hearth in House 2 likely represents cleaning activity, which, along with the charcoal identified in several cores, supports that burned matter was dumped across the site.

Fuel selection is commonly guided by the availability of materials. A viable supply of wood is lacking on the Sub-Arctic tundra, but, during large scale fall caribou hunting, caribou bone provided an alternative fuel source. Crystallinity index, C/P values, and organic matter, collagen, and phosphate peak changes for excavated caribou bone indicate high intensity burning (Fig. 4; Table 2). Unburned samples from dwelling floors were well preserved, demonstrating the diagenetic alterations recorded for hearth samples were caused primarily by burning. The appearance of sharp peaks at 632, 1090, and 962 cm⁻¹ in the spectra with the highest Cl values demonstrate that some samples are calcined and were burned at temperatures exceeding 700 °C (Thompson et al., 2009; Weiner, 2010) (Fig. 4). An experimental heating study in a stållo pit-house reconstruction demonstrated that in outdoor conditions around ~10 °C, a birch wood fire with a core temperature of 437.7 °C produces an ambient indoor temperature ranging between 13.2 and 26.9 °C (Liedgren and Östlund, 2011). Hearths used in the tested Ikirahak dwellings reached higher core temperatures, indicating the houses were well heated, which would not be necessary during warmer months. High temperatures were required because the dwellings were occupied during cold seasons. Maintaining hearths at temperatures exceeding 700 °C would have been an inefficient use of limited fuel during warmer months.

There is no evidence for the use of outdoor hearths. The spatial extent of montgomeryite suggests authigenic formation from widespread refuse, not from ash in discrete, primary hearth areas. There are traces of charcoal in some soils from across the site, but overall, the soils are lacking disorganized calcite, carbonate hydroxyapatite, and hematite. Moreover, Hodgetts et al.’s (2011) magnetometer survey of the site did not detect any signs of burning outside of dwelling contexts. It would have been an inefficient use of sparse fuel to maintain outdoor fires during colder seasons, as these hearths would have been poorly protected against wind and moisture, and they would have required significantly more fuel to produce a sustainable amount of heat. Hearth features were only established inside dwellings, supporting a cold season occupation and providing insight into the use of household rather than communal economic units. Using the frequencies and sizes of hearths and middens to define socio-economics, however, first requires an understanding of feature contemporaneity.

Bone burning, widespread chemical alterations of soils, investments into building pit-houses, and excavated hunting and hide
processing equipment at Ikirahak indicate low residential mobility and repeated occupation. We propose that this tundra-based site functioned as a late fall caribou hunting base camp and processing facility, which is a type of settlement behaviour considerably different from that documented within the Beverly Caribou Range. It also diverges radically from patterns documented for their Chippewyan descendants, who visited the tundra in small, highly mobile groups during summer (Hearne, 1958). Our model contends that southbound caribou herds would have been intercepted at water crossings, such as those identified around Ikirahak Island, during early and mid fall to build surpluses of hides and meat. Housing and clothing a group of families would take several hundred hides, and given the thin, supple nature of caribou leather, people would have to replace their products regularly (trimoto, 1981; Thompson, 1994). Hides were in the best condition for these purposes during fall, after warble fly damage healed, when aggregated caribou were moving south (Hearne, 1958). Sites like Ikirahak were not rapidly abandoned immediately after the hunting season. People stayed into the late fall/early winter to dry meat and prepare hides. Fresh meat would have supported the group during their stay at the settlement, while dried meat would have been prepared and stored for the long trip to the southern forest and for supplementing diets throughout winter. We are currently investigating the impacts of meat and hide processing and storage using X-ray fluorescence, inductively coupled plasma—mass-spectroscopy, gas chromatography, and FTIR analyses of soils and reference materials.

6. Conclusions

This research contributes to demonstrating that FTIR is an accessible, rapid, and cost-effective part of the micro-archaeological tool-kit, and that it is valuable in reconstructions of functions and organizations of spaces at hunter-gatherer settlements. The rapidity and low cost of analyses make the technique useful for identifying areas on sites for further, more complicated and expensive analyses. In our case study, there are no spatially patterned concentrations of authigenic phosphates, supporting the argument that faunal elements from spatially organized middens have not been buried, completely decayed, and reprecipitated. Excavated unburned bones are well preserved, verifying that a buried bone midden would likely survive in this environment. We argue that disordered montomereyite formed from the diagenesis and reprecipitation of carbonates, phosphates, and magnesium added to the system by the dispersal of ash and caribou processing residues. Additional research using experimental deposits, lysisimic, micromorphology, and electron microprobe will help clarify the formation processes and behaviour of authigenic phosphates on open-air hunter-gatherer sites.

Our results support the use of C1, C/P, calcite, and carbonate hydroxylapatite for distinguishing fuel sources in hunter-gatherer hearths. Several excavated bones exhibit high intensity burning, supporting the arguments that bone was used as fuel and that the site was occupied during a cold season. Additional experimental research concerning bone burning/decomposition and mineralization specifically in caribou bone is necessary, given that ratios of organic and mineral components vary across species, age, and habitat. Such variations may influence measurements of crystallinity and burning. Infrared spectra also have signatures for disorganized calcite and carbonate hydroxylapatite in two of the three tested pit-house hearths, indicating the use of both wood and bone for fuel. Based on the absence of hematite, disorganized calcite, and carbonate hydroxylapatite in soils from across the settlement, hearth features were exclusive to dwellings. The use of semi-subterranean dwellings at the Ikirahak site, a housing type previously unidentified in Taltheilei archaeological record, suggests that people occupied this area during a cold season. Visible and chemical traces of burned bone in hearth features support this argument. Bone provided a viable fuel alternative on the tree-less tundra during late fall stays, which we argue is one of the primary reasons the Ikirahak site lacks a bone midden. A late fall occupation of a tundra-based site indicates that Taltheilei settlement strategies were more dynamic than previous research has recognized.

Acknowledgements

We thank the Social Sciences and Humanities Research Council of Canada, the Department of Indian and Northern Affairs Canada, International Polar Year Canada, and the University of Calgary Department of Archaeology for funding our project. We thank Wade White from the University of Calgary Department of Chemistry for FTIR training, Derek Wilson from the University of Calgary Department of Geography for PSA training, Calla McNamee and Howard Cyr from the University of Calgary Department of Archaeology for their micromorphology research, and Sean Pickering from the University of Calgary Department of Archaeology for leading excavations and lithic analyses. We also thank our reviewers for their helpful comments.

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