Reconstructing palaeoenvironments using variations in the isotopic compositions of bison tooth enamel carbonate from Saskatchewan archaeological sites

A Thesis Submitted to the College of Graduate Studies and Research in Partial Fulfillment of the Requirements For the Degree of Master of Arts in the Department of Archaeology University of Saskatchewan Saskatoon

By

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ABSTRACT

Lack of calibrated instruments and written records prior to European contact in North America has forced palaeoclimatic researchers to develop various proxies capable of reconstructing ancient environments. Stable isotope analysis of tooth enamel of large terrestrial herbivores has increasingly become a creditable method of determining the ancient environments which these large mammals occupied during life. Archaeological evidence indicates human inhabitants of the northern Great Plains relied heavily on bison procurement throughout much of the Holocene. Because of this correlation, stable isotope analysis of bison tooth enamel has the capability of informing on palaeoenvironmental conditions which these ancient cultural groups occupied for the last 10,000 years on the northern Great Plains.

Decades of research has provided evidence that stable isotope analysis of tooth enamel of large bodied herbivores (e.g. bovids) has the potential to be used as a proxy for reconstructing palaeoclimate, palaeoecology, foraging strategies and herd behaviour. Oxygen ($\delta^{18}O$) isotope ratios are used as a proxy to track the meteoric hydraulic cycle (i.e. precipitation), which in turn is driven by local surface temperatures. Carbon ($\delta^{13}C$) isotope ratios have the ability to indicate photosynthetic pathways used by plant species, thus indicating local terrestrial plant cover. Dietary intake of water ($\delta^{18}O$) and food ($\delta^{13}C$) are associated with isotopic signals which are recorded in the tooth enamel of a bison during amelogenesis (tooth enamel formation). Once tooth enamel is formed it never remodels; therefore, isotopic ratios recovered from fossil enamel become an archive of dietary consumption. In general, $\delta^{18}O$ isotope ratios are used to determine surface water and surface temperature conditions, whereas $\delta^{13}C$ isotope values are used to indicate the abundance of C$_3$ to C$_4$ grasses consumed during an animal’s life.

This study analyzes stable isotope ($\delta^{18}O$ and $\delta^{13}C$) ratios obtained from fossil bison enamel associated with archaeological sites in the northern Great Plains (Saskatchewan) region. The purpose of this study is to create a comparative model used to indicate ancient seasonality and palaeoenvironmental conditions over a 9,000 year period in the Holocene. A total of eight archaeological sites were examined, with each site representing a distinct time period and an affiliated human culture. In addition, isotope ($\delta^{18}O$ and $\delta^{13}C$) ratios recovered from tooth enamel was compared to isotope ($\delta D$ and $\delta^{13}C$) values previously (Leyden 2004) examined from bone collagen of bison remains from the same archaeological sites.
Results of this study demonstrates that original isotopic values from consumed water ($\delta^{18}$O) and food ($\delta^{13}$C) from archaeological bison tooth enamel reflects seasonal changes for an approximate 18 month period. Further, results from this study also indicate that several climate and plant ecology changes occurred in the Saskatoon, Saskatchewan region over the last 9,000 years. Episodes of climate warming and cooling have been inferred by changes in $\delta^{18}$O ratios at different time periods of the Holocene. Similarly, significant differences are also detected in $\delta^{13}$C values from different archaeological sites, inferring that bison populations consumed various abundances of C$_4$ grasses at different time periods. In addition, evidence from this study has indicated that stable isotope ratios from enamel ($\delta^{18}$O) and collagen ($\delta$D) from the same archaeological site, for the purpose of inferring climate conditions, demonstrate differing data for several time periods and close correlations for others. On the contrary, $\delta^{13}$C from both tooth enamel and bone collagen from each archaeological site produce comparable data which were used to measure the abundance of C$_4$ grasses consumed by bison population during particular time periods.
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CHAPTER ONE: INTRODUCTION

1.1 Introduction

Archaeological research on the Great Plains has provided substantial evidence that human and bison populations lived coevally throughout the Holocene epoch. The earliest occupants of the Great Plains, often referred to as Palaeoindian groups, hunted large mammal species such as mammoths, mastodons, bison and perhaps camels and horses. These large mammal species remained the predominant game for Palaeoindian hunters prior to the end of the Pleistocene, around 11,000 years ago (Frison 1998). After or close to the extinction of these megafauna species, Palaeoindian groups focused on the procurement of bison (Geist 1996; Frison 1998). It is from this early period until European contact that indigenous groups relied heavily on the bison, both economically and socially. Bryan (2005:40) states “seldom in the history of Earth has a single animal species had such drastic influence on human lives”.

Economically, the bison has been referred to as a “tribal department store” (Krech 1999:128), a comment which suggests that the Plains people used the entire bison carcass to benefit their livelihood. As a food source, the bison made up a large portion of the Plains people’s diet, including meat, fat, bone grease and marrow. As raw materials, bison hides were processed to provide shelter and clothing; bison bones were crafted into tools; numerous bison organs such as the bladder, horns, scrotum and paunch were used as containers; bison tendons (or sinew) and wool were manufactured into cordage and string; and bison dung was used to fuel fires (Roe 1970; Foster and MacLaren 1992; Bryan 2005). With its ability to meet all these fundamental needs, it is logical to suggest that the Plains people would have not been able to flourish on the Great Plains without the bison.

With so many commodities of primary importance derived from the bison, Plains cultures developed social as well as ideological systems based on the massive headed, high humped ungulate. Settlement patterns and subsistence strategies of Great Plains cultures were largely based on bison annual migrations and bison behaviour (Roe 1970; Foster and MacLaren 1992). Large social gatherings came in the form of communal hunts, where numerous groups coalesced to form a regional band (Frison 1973; Arthur 1975). As Martin (1978:113) put it “perhaps one of the most stunning ways to phrase the Indian conception of hunting is to say that it is nothing
short of a ‘holy occupation’”.

At the time of European contact, the American Bison (*Bison bison bison*) was flourishing with an estimated population of 30 to 50 million (Brink 2008). As the largest ruminant occupying the Great Plains, the bison became a walking target for European settlers. Between the collision of the Old World and New World, and the introduction of the European style market economy, mass hunting brought the bison to near extinction. The catastrophic slaughter of the bison not only affected the entire ecosystem of the Great Plains but it also crushed the native societies of those who hunted the majestic beast. Bison hunts were supported by the federal authorities who saw the destruction of the bison as the key to Native American acceptance of reservation systems (Isenberg 2000). Only a few Euroamericans saw the bison as the symbol of the American frontier and thus developed a mission to conserve this living artifact of the New World. With the bison gone, over 10,000 years of Plains peoples’ history ceased to exist.

Modern research methods and techniques such as stable isotope analysis have become an important tool used for reconstructing climate and environmental conditions prior to the collection of instrumental data (e.g. use of thermometers to collect comprehensive temperature records). Pioneer studies (e.g. DeNiro and Epstein 1978; Bryant and Froelich 1995; Passey et al. 2005a) have directly correlated with source specific food and water sources which are digested during an animal’s life leaving isotopic signatures in the animals tissues indicating the relationship between an organism and its ecosystem. Because of this effect, tissues such as tooth enamel which survives long after the animal is dead, can be isotopically analyzed for the purpose of reconstructing climate and environmental settings a particular specimen occupied. Bison continuously form teeth over several years after birth and isotopic values of ingested nutrients are secreted in the enamel of a developing tooth (Passey et al. 2005a). Once tooth enamel is completely mineralized, it does not remodel; thus the isotope values obtained from a bison tooth can be used as a climate and environmental proxy of the time over which the tooth was formed (Gadbury et al. 2000). Bison teeth are commonly found in prehistoric archaeological sites on the Great Plains and are often directly associated with human material remains; therefore, isotopic values obtained from analyzed teeth are an archive of palaeoclimatic and palaeoenvironmental conditions experienced by past human populations.
1.2 Objectives of this Thesis

The main objectives of this study are to expand our knowledge of the palaeoclimatic and palaeoenvironmental history on the northern Great Plains during the Holocene as well as to further our understanding of what information we can obtain from the stable isotope analysis of various bison biological tissues (i.e. enamel and collagen). In order to achieve these objectives, several criteria will be measured and analyzed. First, the research method of sequentially sampling and isotopically analyzing bison tooth enamel has the potential to provide an environmental proxy which has the capability of identifying seasonal fluctuations associated with temperature (i.e. precipitation) and terrestrial vegetation (i.e. dietary grasses) variations at a scale of a 15 to 18 month time period. Second, the mean oxygen and carbon isotope values obtained from eight fossil bison teeth representing various time periods of the Holocene have been used in a comparative model for the purpose of indicating ancient climate conditions in Saskatchewan over a 9000 year period. Third, mean oxygen and carbon isotope values from tooth enamel carbonate have been compared and contrasted with hydrogen and carbon isotope values of bone collagen obtained from the same bison populations of eight prehistoric archaeological sites located in southern Saskatchewan for the purpose of indicating whether similar palaeoenvironmental conditions are indicated by these two types of bison skeletal tissues. Finally, mean oxygen and carbon isotope values obtained from bison teeth and hydrogen and carbon isotope values of bison bone collagen from eight prehistoric archaeological sites located in southern Saskatchewan have been measured and compared against aquatic palaeoenvironmental proxy studies previously and independently conducted in this study for the purpose of indicating whether similar palaeoenvironmental conditions are detected from terrestrial and aquatic proxies.

1.3 Thesis Structure

Chapter two of this thesis is the literature review and includes various topics which are vital in understanding the materials and methods used in this study including: hypsodont teeth, bison teeth in archaeological research, isotopic reservoirs in tooth enamel, general characteristics of stable isotopes, isotope fractionation processes, stable oxygen and carbon isotope analyses, bison foraging and herd behavior, and a brief overview of the physical environment of the study area. Chapter three examines the results of oxygen and carbon isotope data obtained from the
bison teeth utilized in this study. Results are used in a fourfold fashion: First they are analyzed at an individual level, where sequential samples from a specimen were examined for the purpose of detecting seasonal changes in temperature and dietary food sources during the development of a bison third mandibular molars; secondly, mean isotopic data from all eight teeth are compared and contrasted to infer changes of palaeoclimatic and palaeoenvironmental conditions on the northern Great Plains region over the last 9,000 years; thirdly, mean isotopic data from bison tooth enamel (δ¹⁸O, δ¹³C) and collagen (δD, δ¹³C) from the same eight archaeological sites are compared and contrasted to determine whether isotopic analysis data from various biological tissues indicate the same palaeoclimatic and palaeoenvironmental conditions; fourthly, palaeoenvironmental data from terrestrial palaeoenvironmental proxies (isotope analysis on bison teeth and bone collagen) and aquatic proxies (lake cores) are compared and contrasted to determine if both palaeoenvironmental reconstruction methods determine similar conditions in the study area.

Chapter four is the concluding discussion of this study. Discussion will include a brief summary of our findings, potential problems, as well as the focus of future research which can potentially expand our knowledge of the palaeoclimatic and palaeoenvironment of the northern Great Plains. Finally, Appendix A includes brief discussions of the archaeological sites which yielded the teeth collected and analyzed in this study. Appendix B includes the radiocarbon dates associated with these archaeological sites. Appendix C is an overview of the techniques and methods used in this study.
CHAPTER TWO: LITERATURE REVIEW

2.1 Development of Hypsodont Teeth

Hypsodont (high-crowned) cheek teeth occur in many herbivorous mammal lineages which have abrasive diets. Hypsodont teeth may be described as having a well-developed crown which extends far above the gum-line or root of the tooth. Tooth abrasion occurs through the consumption via grazing of silica-bearing grasses, as well as grit particles adhering to ingested vegetation. Additional crown height thus provides an increase of tooth material which is worn down throughout an animal’s lifetime. The evolution or development of hypsodont dentition in herbivorous mammals is often cited as a classic case of animals adapting to their environment.

Palaeontological records from North America, Africa, South America, and Asia during the early Miocene (about 24 myr ago) provide evidence that most herbivorous mammals had brachydont (low-crowned) or mesodont (medium-crowned) molar teeth (Cerling et al. 1999; Jernvall and Fortelius 2002; Fortelius et al. 2006). Environmental conditions at this time, as indicated by carbon isotope analyses of tooth enamel (Cerling et al. 1997) and soil carbonates (Janis 1998), reflect high CO$_2$ concentrations and moderate climates (Cerling et al. 1999). Terrestrial flora during the early Miocene was thus a “C$_3$-dominated world” characterized by mixed forest and grassland habitat (Cerling et al. 1999).

At the late Miocene (about 11 myr ago), major climatic changes occurred throughout much of the world, introducing a trend to toward cooler, more arid conditions (Janis 1998; Fortelius et al. 2006) and a decrease in CO$_2$ atmospheric concentration levels (Cerling et al. 1999). These conditions produced low-biomass environments better suited for plants which use the C$_4$ photosynthetic pathway (Cerling et al. 1997 and 1999). It is at this time that C$_4$ grasses became progressively more dominant in plains and savanna type environments throughout most of the world (Cerling et al. 1997, Janis 1998; Cerling et al. 1999).

The herbivorous mammal transition from brachydont to hypsodont tooth form in the palaeontological record strongly reflects the transition from C$_3$ plant to C$_4$ plant dominated environments (Cerling et al. 1997; Jernvall and Fortelius 2002; Theodor 2002; Fortelius et al. 2006). Because C$_4$ grasses are often fibrous and have a high silica content, hypsodonty development allowed large herbivorous mammals to increased their range of available resources.
By the end of the Miocene (5 to 8 myr ago), hypsodont mammals greatly outnumbered those with brachydont dentitions in most geographic locations (Theodor 2002).

2.2 Bison Teeth in Archaeological Research

Prior to the 1960's and early 1970's, very little emphasis was placed on animal bone found in archaeological sites (Thomas 1996). Lack of formal training in anatomical and species identification, as well as the absence of quantitative methods made many regard the study of animal remains unproductive and untrustworthy (Thomas 1996). The birth of processual archaeology introduced a more scientific method of analyzing archaeological assemblages. Processualism incorporated the aims of scientific method to archaeological excavations by formulating hypotheses and then proceeding to answer them (Renfrew and Bahn 2004). By taking this approach, archaeologists began incorporating explanatory frameworks such as subsistence strategies and settlement patterns (Thomas 1996). This scientific transition led to the increased importance of understanding animal remains in archaeological sites, thereby; creating a new specialty we now call zooarchaeology.

Bison teeth have played a fundamental role in Great Plains archaeology, specifically when found in large quantities such as at kill and butchering sites. Numerous studies (Frison and Reher 1970; Frison et al. 1976; Frison and Stanford 1982; Frison and Todd 1987) have been conducted on tooth eruption patterns and dentition wear of bison teeth for the purpose of determining at age and season of mortality. Historic and extant bison herds generally begin calving in the latter part of April and the first part of May (Frison and Reher 1970). Due to the reproductive synchronization of bison, tooth eruption patterns of calf mandibles can be used to age an individual specimen and if found in archaeological context, determine the seasonality of the site. Age determination of adult bison is done through the analysis of teeth wear. Because of their abrasive diets, bison exhibit occlusal wear on their cheek teeth which decrease in height overtime. Tooth wear analysis is done through the measurement of the enamel heights of the metaconid cusp of molar teeth. Measured metaconid values are used in a comparative model against a reference collection of bison specimens of known ages at death (Frison and Reher 1970).

Nutrient sources which are digested during an animal’s life leave isotopic signatures,
indicating the relationship between an organism and its ecosystem (DeNiro and Epstein 1978; Bryant and Froelich 1995; Passey et al. 2005b). Put another way, isotopic values retained in animal tissues are directly linked to behaviour, physiology, and most importantly, diet (Koch 1998). For this reason, stable isotope analysis (SIA) has become a very useful tool in many disciplines dealing with the natural world (e.g. palaeontology, biology, geology, ecology, archaeology). Bison teeth continuously form over several years. Isotopic values of ingested nutrients are secreted in the enamel of a developing tooth. Once tooth enamel is completely mineralized, it does not remodel; thus, the enamel becomes an isotopic reservoir of an animal’s environment at the time during which its teeth formed.

2.3 Isotopic Reservoirs in Tooth Enamel

Pioneer studies (DeNiro and Epstein 1978; Vogel 1978; Longinelli 1984; Luz et al. 1984; DeNiro 1987; Schwarz and Schoeninger 1991; Quade et al. 1992) have focused on the relationship between the stable isotope values of animal tissues and their diets. At a molecular level, food and water contain various proportions of the different stable isotopes of carbon, oxygen, nitrogen and hydrogen (Hall 1961). In most cases, the stable isotopic compositions of these elements are source specific; thus, they indicate the conditions and reactions that produced the changes. Evidence from these pioneer studies has indicated that individuals of the same species of an animal have different isotope values when ingesting different diets. Conversely, different individuals of the same species fed the same diet have similar isotope values, thus indicating the ingested diet is the primary source of an animal’s isotopic value. Such studies scientifically provide evidence that what you ate can be revealed through isotope analysis, “you are what you eat” (Katzenburg 1991).

Both organic (e.g. collagen) and inorganic (e.g. enamel) animal tissues found in archaeological sites have been used in dietary isotope studies; each tissue type has positive and negative attributes. Initially, collagen was the tissue of choice in archaeological studies because of the slow “turnover” rate (10-20 years) of its isotopic compositions (Hall 1961; Stenhouse and Baxter 1979). This slow “turnover” rate produced isotopic values which averaged an animal’s dietary intake throughout much of its life. Unfortunately, too many studies (e.g. Lee-Thorp and van der Merwe 1987; Wang et al. 1994; Ayliffe et al. 1994) conducted on bone collagen have produced many aberrant isotopic values that have been irreparably altered by digenetic effects.
Further, it is currently understood that dietary information obtained from organic and inorganic tissues do not retain equivalent isotopic data and as such provide different perspectives (Sponheimer and Lee-Thorp 1999). Organic material such as bone collagen isotopically reflects only ingested protein in the diet and not the entire integrated diet (Ambrose and Norr 1993; Tieszen and Fagre 1993; Howland et al. 2003; Jim et al. 2004; Kellner and Schoeninger 2007; Warinner and Tuross 2009).

Although tooth enamel only represents a snap shot ranging from months to several years of an animal’s dietary intake, many positive attributes have made it the current tissue of choice in dietary isotopic studies. Unlike collagen, isotopic values obtained from enamel represent an animal’s entire diet. Enamel is composed of large tightly packed apatite crystals which are highly organized, making enamel less susceptible to post-depositional isotope exchange via diagenesis than organic material (Lee-Thorpe and van der Merwe 1987; Quade et al. 1992; Passey and Cerling 2002). Because enamel forms in a sequential pattern with a fast deposition rate, it is a useful tissue to research small-scale environmental changes and short-term seasonal fluctuations. Also, enamel exists on the exterior of the tooth making it easy to reach limiting destruction of the tooth during sampling.

Stable isotope analysis studies have been conducted on the enamel of hypsodont animals for the purpose of reconstructing ancient seasonal variations in climate, diet, and animal behaviour throughout the animal’s life (Fricke and O’Neal 1996; Fricke et al. 1998; Kohn et al. 1998; Sharp and Cerling 1998; Gadbury et al. 2000; Fox and Fisher 2001; Passey and Cerling 2002; Passey et al. 2005b; Zazzo et al. 2005). Although teeth come in all shapes and sizes, they all share the three basic dental tissues, enamel, dentine, and cementum (Hillson 1986; Aiello and Dean 1990). Amelogenesis is the process of tooth enamel formation and occurs throughout the development of the tooth (Suga 1982). Enamel consists mainly of calcium and phosphate minerals in the form of hydroxyapatite which is often also referred to as bioapatite ($\text{Ca}_{10}(\text{PO}_{4})_{6}(\text{OH})_{2}$) (Suga 1982; Hillson 1986; Aiello and Dean 1990; Balasse 2002). In its mature state, the physical compositions of enamel includes inorganic material (96-97%), organic material (0.04-0.9%) and water (3.6-2.1%) (Hillson 1986). Due to its high content of inorganic material notably, apatite crystals and sturdy organic proteins such as cementum, enamel is the hardest known biologically formed substance (Suga 1982; Aiello and Dean 1990).
Amelogenesis is divided into two formation steps, the first step being the matrix production or secretory stage, followed by the maturation stage (Suga 1982; Hillson 1986; Aiello and Dean 1990; Zazzo et al. 2005). The matrix production stage is predominantly characterized by the formation and deposition of organic (30%) rich material along with apatite crystallites called amelogenin or enamel matrix protein (Hillson 1986; Zazzo et al. 2005). The maturation stage includes the incremental replacement of the deposited amelogenin with inorganic apatite (Hillson 1986). Both formation steps of amelogenesis are produced by specialized cells called ameloblasts, which accumulate into sheet-like structures (Hillson 1986; Aiello and Dean 1990; Passey and Cerling 2002). Remaining active through the entire process of amelogenesis, ameloblasts secrete enamel at the enamel-dentine junction (EDJ) and continue to push the matrix that they lay down outward until the entire enamel thickness is reached (Aiello and Dean 1990). Ameloblasts form the apex of the crown initially and progressively move downward to the tooth’s cervix (Hillson 1986; Passey and Cerling 2002; Zazzo et al. 2005).

Every enamel layer that is secreted during amelogenesis produces two types of incremental growth lines, cross striations and brown stria of Retzius, which can be seen at microscopic levels (Suga 1982; Hillson 1986; Aiello and Dean 1990; Passey and Cerling 2002). Cross striations are the smaller incremental lines and are produced by variation in mineralization which represents circadian deposits. Brown stria of Retzius are incremental growth lines that represent the enamel matrix-forming front. The stria of Retzius are produced by the slowing of matrix secretion and the start of mineralization and generally occur at equal periods throughout amelogenesis.

Mammals continuously form tooth enamel over several years (Suga 1982; Hillson 1986; Aiello and Dean 1990; Gadbury et al. 2000). Studies have shown (Gadbury et al. 2000; Zazzo et al. 2005 and 2006) that bovines form molars in a constrained time period. Adult bison exhibit three molars, M₁, M₂ and M₃, in each dentition quadrant. M₁ begins forming en utero and is fully mineralized several months after birth; M₂ begins formation around a month after birth and is complete around a year later; M₃ begins forming at around nine months after birth and continuously develops until the animal is ca. 2 years. Once tooth enamel is fully mineralized, it does not remodel, making it an archive for carbon and oxygen isotopic composition of ingested food (carbon) and water (oxygen) during enamel mineralization (Balasse 2002; Passey and
Cerling 2002; Passey et al. 2005a; Zazzo et al. 2006). Put another way, high crowned teeth such as a bison molar, will form, from crown to root, an isotopic time-series of dietary and environmental changes several years of an animals’ early life. Therefore, M₃ of a bison represents the dietary intake of that individual specimen for roughly a 15-to-18 month period.

2.4 General Characteristics of Stable Isotopes

Around a century ago, chemists discovered that all atoms of a particular element are not exactly the same. Atoms of a specific element share the same atomic number (number of electrons and protons) but may have variable numbers of neutrons within the nuclei. The atomic mass (number of protons and neutrons) thus varies in a given element, with different numbers associated with isotopes of that element different element. The majority of naturally occurring elements contain more than one isotope (Krauskopf and Bird 1995). Particular elements which have more than one isotope are referred to as isotopic species (Schwarcz and Schoeninger 1991)

Isotopes exist in two forms, those that are stable and those that are not. Approximately 1200 unstable isotopes are known to exist in nature (Hoefs 1980). Prototypic unstable isotopic species (parent atoms) radioactively decay overtime, transforming into nuclei of other elements (daughter products) (Krauskopf and Bird 1995). Numerous radioactive isotopes decay at a constant rate unaffected by natural processes such as temperature, pressure or chemical reaction (Krauskopf and Bird 1995). Inorganic material such as rocks and soil can be isotopically analyzed (Rb-Sr, U-Th-Pb, K-Ar, Sm-Nd) by the relative concentrations of the daughter-parent isotope ratios, whereas organic material is analyzed (C¹⁴) through the actual amount of radioactivity of a particular element (Krauskopf and Bird 1995). Although the details of radiogenic isotope decay are beyond the scope of this thesis, their instability of radioactive isotopes provides many earth scientists, including archaeologists, with absolute dates.

There are approximately 300 stable isotopes which exist in all known elements (Hoefs 1980). Stable isotopes species of low atomic numbered elements have sufficient proportional differences in atomic mass to react at different rates, resulting in isotopic fractionation (Hoefs 1980; Krauskopf and Bird 1995). Measurement of an element’s isotopic fractionation thus indicates the conditions and reactions that have naturally occurred. Analysis of stable isotope ratios has become an important tool for measuring modern and ancient earth processes such as
temperature, climate and ecology, as well as movement of food and water in modern and ancient biological food-webs. Because of their natural occurrence and differences in the chemical properties of their isotopes, H, C, N and O have become the most commonly used elements in investigating earth processes and biological food-webs (Koch 1998). All four of these elements have isotopic species which vary in atomic mass and abundance (Table 2.1). The lighter isotopic species of the element is generally more abundant than the heavier isotope which tends to be much rarer (Hoefs 1980; Krauskoff and Bird 1995; Walther 2005). Hence, statistical measurements of elements isotopes occur in the form of heavy-to-light ratio.

Table 2.1 Isotopic Species of Four Elements Frequently Used in Investigating Earth Process and Food-Webs

<table>
<thead>
<tr>
<th>Element</th>
<th>Stable Isotope Species</th>
<th>Percent Abundance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>$^1$H</td>
<td>99.986</td>
</tr>
<tr>
<td></td>
<td>$^2$H</td>
<td>0.015</td>
</tr>
<tr>
<td>Oxygen</td>
<td>$^{16}$O</td>
<td>99.76</td>
</tr>
<tr>
<td></td>
<td>$^{17}$O</td>
<td>0.04</td>
</tr>
<tr>
<td></td>
<td>$^{18}$O</td>
<td>0.20</td>
</tr>
<tr>
<td>Carbon</td>
<td>$^{12}$C</td>
<td>98.89</td>
</tr>
<tr>
<td></td>
<td>$^{13}$C</td>
<td>1.11</td>
</tr>
<tr>
<td>Nitrogen</td>
<td>$^{14}$N</td>
<td>99.63</td>
</tr>
<tr>
<td></td>
<td>$^{15}$N</td>
<td>0.37</td>
</tr>
</tbody>
</table>

The data used in Table 2.1 is adapted from Ehleringer and Rundel (1989).

Initial isotopic research indicated that laboratories analyzing the same sample, through the use of an isotope-ratio mass spectrometer, may produce variable measurements (Hoefs 1980). For this reason, arbitrary standards were created for each element as well as an accepted method for representing and reporting SIA content of analyzed samples (Hoefs 1980). The delta-value
(δ) notation is used to express the isotope ratio between the sample and the standard (Hoefs 1980; Spielmann et al. 1990; Krauskoff and Bird 1995; Walther 2005). δ values are followed by the symbol (‰), which stands for per mil (parts per thousand). Due to the fact that the separation values between the sample and standard are very small, cognitively speaking, sample-to-standard ratios are calculated as follows:

\[
\delta \text{ heavy} = \left( \frac{R_{\text{sample}} - R_{\text{standard}}}{R_{\text{standard}}} \right) \times 1000 \text{‰}
\]

Where ‘δ heavy’ is the heavy isotope of the element, \( R_{\text{sample}} \) is the ratio (heavy-to-light) of the sample, and \( R_{\text{standard}} \) is the isotope ratio of the known standard. The 1000 ‰ is present to make the values larger then 0.0001 or so and therefore easier to compare for differences. For example, the isotopic ratio of oxygen in a sample is then given by:

\[
\delta ^{18}\text{O} = \left( \frac{^{18}\text{O} / ^{16}\text{O}}{^{18}\text{O} / ^{16}\text{O}} \right)_{\text{sample}} - \left( \frac{^{18}\text{O} / ^{16}\text{O}}{^{18}\text{O} / ^{16}\text{O}} \right)_{\text{V-SMOW}} \times 1000 \text{‰}
\]

A more positive \( \delta ^{18}\text{O} \) value (e.g. 22‰) would indicate enrichment in the heavy isotope relative to Vienne Standard Mean Ocean Water (V-SMOW), which is the standard for hydrogen and oxygen, whereas a negative value (e.g. 12‰) would represent depletion in the isotope.

### 2.5 Isotope Fractionation Processes

Variations in the physical and chemical properties of an element’s isotopes may permit isotope fractionation, or some separation of heavier from lighter isotopes of the same element in natural occurring processes, resulting in products with distinct isotope ratios (Koch 1998). Isotope fractionation is largely a result of the different vibrational frequencies associated with the lighter and heavier atoms found in a molecular or chemical structure (Krauskoff and Bird 1995). Atoms of the lighter isotopic species store less potential energy than those that are heavy (Walther 2005). For this reason, atoms of the heavy isotopic species have a slightly stronger bond than those of the lighter variety and therefore, harder to separate. Therefore, elements which have isotopes with the largest relative mass differences will show greater fractionation.
than elements containing a smaller mass difference.

Measurable differences of isotope fractionation generally occur in elements with atomic masses of less than 40 (Hoefs 1980; Koch 1998). During vibrational frequency of particles, which is caused by an increase in energy (i.e. the sun), isotopic species which have ionic and metallic bonds (i.e. elements with heavy atomic masses) do not share electrons with other elements (Walther 2005). This results in bonds that remain unaltered by mass dependent effects, thus creating minimal isotopic fractionation. In contrast, isotopes of most elements with low atomic masses are covalent bonds along which electrons are shared. This results in mass dependent vibrational frequencies, creating measurable fractionation effects when molecules containing such bonds are involved in chemical reactions. Importantly, fractionation effects are largely temperature dependant (Hoefs 1980; Krauskoff and Bird 1995; Walther 2005). As temperatures increase, the separations between an element’s isotopic species become less pronounced. This occurs because higher temperatures increase vibrational and rotational frequencies resulting in excited atoms. As temperatures rise, the bond strength of the lighter and heavier species becomes similar, thus decreasing the importance of mass dependent effects.

There are two processes which produce isotope fractionation, *thermodynamic equilibrium isotope effects* and *kinetic isotope effects* (Hoefs 1980; Koch 1998; Walther 2005). Thermodynamic effects work on a bidirectional basis and are a result of mechanisms that are dependent on the physical properties of molecules involved (Krauskoff and Bird 1995; Koch 1998). For example, during evaporation, the light oxygen isotope ($^{16}$O) is enriched in vapor water and depleted in liquid water. On the other hand, the oxygen isotopic ratio of precipitation is enriched in the heavy isotope ($^{18}$O). The reason this fractionation occurs is because H$_2$O molecules containing the lighter O isotope are more easily excited than those containing the heavy isotope and therefore the former have a higher vapor pressure (Krauskoff and Bird 1995).

Isotope fractionation via thermodynamic effects also occurs through the exchange reactions of two or more species in the same physical phase (Walther 2005). In an example taken from Walther (2005), when CO$_2$ gas containing only $^{16}$O is mixed with water vapour containing only $^{18}$O, exchange between the two species will occur as follows:

\[ \frac{1}{2} \text{C}^{16}\text{O}_2 + \text{H}_2^{18}\text{O} = \frac{1}{2} \text{C}^{16}\text{O}_2 + \text{H}_2^{16}\text{O} \]
until equilibrium is reached. Although equilibrium is reached in the isotopic ratios of $^{18}$O/$^{16}$O in CO$_2$ and H$_2$O, fractionation effects remain unique due to the difference in bond strength between the two compounds (Krauskoff and Bird 1995).

Kinetic isotope effects are dominated by differences in reaction rates among isotopes and generally occur in transport processes (Hoefs 1980; Koch 1998). Because less energy is needed to excite a lighter isotope than a heavier one, chemical reactions initially produce an enrichment of the lighter species. Equally important is the rate at which isotope reactions change in various materials and temperatures (Krauskoff and Bird 1995). For this reason, the ratio of rate constants for the reaction of the light and heavy isotope species may be measured for the degree of fractionation effects (Hoefs 1980).

Because kinetic effects are often constant and predictable, isotopic ratios associated with biological and ecological material have the ability to reveal the amount of time it has taken to occur and the origin of a particular biochemical reaction. Further, kinetic effects may potentially indicate the diffusion rate of gases. Put another way, isotopic analysis has the potential to indicate chemical and physical reactions which occur in nature such as photosynthesis, various phase-changes (hydraulic cycle), and metabolic rates of organisms such as ruminants (Koch 1998; Walther 2005).

The core of this thesis is based on the variations of carbon and oxygen isotope ratios. Research using carbon isotopic variations has focused on several continental and oceanic settings, including photosynthetic pathways used by land plants, reconstructing CO$_2$ levels in the atmosphere, correlations between land and marine events, as well as numerous dietary studies of omnivorous and herbivorous animals. Oxygen isotopic variations have been used to reconstruct continental and oceanic temperatures, as well as trace the hydraulic cycle. Because land biota are directly influenced by the isotopic values of their surrounding environment, ancient skeletal remains can be isotopically analyzed for the purpose of reconstructing inhabited environments.

2.6 Stable Oxygen Isotope Analysis

The earth consists of four major subsystems: the lithosphere, biosphere, hydrosphere, and atmosphere. Oxygen is the most common element found throughout these four subsystems. Oxygen occurs in three stable isotope forms, oxygen-16 ($^{16}$O), oxygen-17 ($^{17}$O) and oxygen-18
(\textsuperscript{18}O). \textsuperscript{16}O (99.76\%) is the most abundant form found throughout the earth’s subsystems, followed by \textsuperscript{17}O (0.038\%) and \textsuperscript{18}O (0.200\%) (Hoefs 1980; Walther 2005). Because of their atomic weight and abundance differences, \textsuperscript{18}O/\textsuperscript{16}O fractionation values occur in different environmental conditions, predominantly reflecting variations in surface temperature. \textsuperscript{18}O/\textsuperscript{16}O proportions therefore can be used to trace water movement through the hydrosphere (Dansgaard 1964; Rozanski et al. 1993).

The hydrosphere consists of water in three physical states; liquid, gas and solid. The three states form the hydraulic cycle, which is powered by the sun. The sun (i.e. insolation) produces the energy required for these states to change. Change of state occurs by the absorption and release of energy. During the physical state change, oxygen isotopes undergo fractionation processes (Dansgaard 1964). The proportion of \textsuperscript{18}O to \textsuperscript{16}O is used to measure the degree of fractionation which occurs in the hydraulic cycle. This proportion is represented by $\delta$\textsuperscript{18}O which is reported on a parts per thousand scale represented by per mil (‰) (Speilmann et al. 1990). $\delta$\textsuperscript{18}O is calculated using the standard V-SMOW which has a known value of 0.5 ‰ or the standard V-PDB which has a value of -8.00 ‰ (Speilmann et al. 1990).

In the deep ocean reservoir, $\delta$\textsuperscript{18}O values are nearly 0.0 ‰ everywhere due to ideal mixing conditions (Walther 2005). Meteoric water (precipitation and evaporation) derived from the ocean may undergo various degrees of fractionation which alter the physical properties of oxygen. There are three main processes, altitude, distance (from the oceanic reservoir) and latitude which affect the oxygen isotope ratio. All of these processes are temperature dependant and are a result of air mass cooling (Dansgaard 1964). For this reason, temperate and boreal forest regions of the earth’s mid latitudes, show a strong correlation between the mean annual temperature (MAT) and the mean annual meteoric water \textsuperscript{18}O values (Koch 1998).

Evaporation occurs in non-equilibrium conditions where the air mass is vapor undersaturated. Evaporation which occurs at higher latitudes and therefore lower temperatures will have $\delta$\textsuperscript{18}O values more negative than evaporation occurring at lower latitudes and therefore higher temperatures). The reason for this is that at higher latitudes the sun’s energy is less intense lowering rates of evaporation in comparison to lower latitudes. Because \textsuperscript{16}O is the lighter of the two oxygen isotopes, water molecules containing it evaporate first, leaving the heavier \textsuperscript{18}O isotopes behind. In cold regions, where little evaporation takes place, the ratio difference
between $^{18}$O and $^{16}$O is significant indicating low or depleted $\delta^{18}$O values.

Once in the atmosphere, water in the form of vapor will move across the continental land masses in air mass systems until near equilibrium conditions of vapor-saturation are reached, condensing into precipitation. The formation of precipitation modifies the isotopic composition of oxygen. Initial precipitation will have enriched $\delta^{18}$O values because the heavier isotope will fall out of the air mass first. As the air mass continues to move inland away from the ocean, $\delta^{18}$O values in the atmosphere become lighter, a process known as Rayleigh distillation (Walther 2005). For this reason, higher latitude locations (lower temperature) have a greater isotopic discrimination factor than those of lower latitudes (warmer temperatures). Therefore, seasonal temperature changes on the northern Great Plains should yield significantly variable $\delta^{18}$O values. Because O and H isotopes in water exist in the same bonds and are similarly affected by the same external process, isotopic values of these two elements in precipitation can be linearly described in the global meteoric water line (GMWL) (see Walther (2005) for detailed discussion).

Local water reservoirs (i.e. lakes, ponds, rivers) in most hydrological settings are recharged by direct precipitation in the form of rain and snow or indirect precipitation such as surface runoff or groundwater (Fritz et al. 1987). If the water reservoirs do not experience significant evaporation, the $\delta^{18}$O values of these recharging sources will generally represent the initial isotopic value of its precipitation origin. Soil water which has entered the lithosphere will initially have a $\delta^{18}$O value similar to the precipitation it derived from. If temperatures increase, soil water may evaporate, therefore enriching the $\delta^{18}$O values of soil water remaining (Gat 1996). The $\delta^{18}$O values of water in the roots and stems of plants generally has a positive correlation with soil water $\delta^{18}$O values, but water located in plant leaves will have enriched $\delta^{18}$O values due to the processes of evapotranspiration (Koch 1998). $\delta^{18}$O values are even more enriched in the leaf water of terrestrial plants where climates are hot and dry (Dongmann et al. 1974; Yakir et al. 1990).

Oxygen isotope variations in/of large (>100 kg) mammals tooth enamel is derived from body water, a combination of ingested drinking water, plant leaf water and metabolic water, generated by the process of food oxidation (Luz et al. 1984; Koch 1998). Among all the animal tissues which contain oxygen isotope variations, tooth enamel is suggested to be one of the best
Mammalian tooth enamel is formed at 37°C and is precipitated in crystalline apatite, which has a strong bond to oxygen (Kohn et al. 1998; Zazzo et al. 2002). Once enamel becomes fully mineralized it does not remodel, therefore recording a permanent δ¹⁸O value with respect to an animal’s body water at the time of mineralization (Stuart-Williams and Schwarcz 1996). Although δ¹⁸O values of tooth enamel generally mirror local water reservoirs, the relationship between the two is not one-to-one (Fricke et al. 1998). The relationship can be described in the form of an equation as:

\[ \delta^{18}O_e = m \delta^{18}O_p + c \]

\(^{18}O_e\) is the value of tooth enamel (carbonate), while \(m\) (0.97 for Bos taurus) and \(c\) (7.28 for Bos taurus) is the slope and intercept, respectively, and are representative of the species in question (see Bryant and Froelich 1995:4524 for various species values). A unity slope represents equality between enamel and precipitation values, whereas a positive slope represents a more positive δ¹⁸O value in enamel than precipitation, this fractionation process is caused by the metabolism and behavior of a particular species (Fricke et al. 1998). Further, the tightly packed apatite crystals of tooth enamel are less susceptible to diagenetic alterations than other less dense skeletal tissues. Because local water composition is temperature and humidity dependant, the oxygen isotope values of tooth enamel are a useful tool for the purpose of monitoring ancient seasonality and climates at global and local scales (Longinelli 1984; Luz et al. 1984 and 1990; Luz and Kolodny 1985; Ayliffe and Chivas 1990; D’Angela and Longinelli 1990; Koch et al. 1998).

2.7 Stable Carbon Isotope Analysis

The primary source of carbon in herbivorous tooth enamel is through food (DeNiro and Epstein 1978; Koch 1998). Numerous feeding experiments (DeNiro and Epstein 1978; Vogel 1978; Quade et al. 1992; Koch et al. 1994; Wang et al. 1994; Morgan et al. 1994; Passey et al. 2002; Zazzo et al. 2005 and 2006) have been conducted on herbivores, for the purpose of indicating a constant \(^{13}C/^{12}C\) ratio correlated with an animal’s diet and skeletal remains. Such studies have shown that the \(^{13}C/^{12}C\) ratio of an animal’s skeletal tissue, including tooth enamel is
enriched 11-14‰ when compared to the actual diet (Koch 1998; Gadbury et al. 2000). Because studies such as these have demonstrated a constant relationship between the $^{13}$C/$^{12}$C ratio of an animal’s enamel diet, stable carbon isotope analysis has become an important tool for reconstructing dietary intake as well as ancient ecology of local environments.

Carbon is one of the most abundant elements in the universe and occurs naturally as two stable isotopes: $^{12}$C (98.89%) and $^{13}$C (1.11%) (DeNiro 1987). On earth, carbon occurs as a trace element and is contained in two main reservoirs: organic matter and sedimentary carbonates. Isotopically, these two reservoirs experience different fractionation mechanisms (Cerling et al. 1993). Organic matter experiences the kinetic effect through photosynthesis, which leads to the depletion of $^{12}$C in the remaining CO$_2$, leaving the $^{13}$C value relatively unaltered (Hasdorf and DeNiro 1985). The proportion of $^{12}$C to $^{13}$C is used to measure the degree of fractionation which occurs in the biosphere and is represented by $\delta^{13}$C (Speilmann et al. 1990). The proportion of $\delta^{13}$C values of a measured sample is measured against the standard V-PDB (Vienna Pee Dee Belemnite Limestone) which has a known $\delta^{13}$C value of 1 to 0‰ (Hutchinson et al. 1998).

Through the kinetic effects of photosynthesis, carbon isotope fractionation has been measured through laboratory plant growth (Park and Epstein 1960). Park and Epstein (1960) also distinguished that each of the three photosynthesis pathways has a distinctive $\delta^{13}$C value.

The Calvin Cycle, also known as C$_3$ photosynthesis occurs in plants which are less sensitive to cold temperatures. Plants using the C$_3$ photosynthesis pathway fix CO$_2$ as a three carbon sugar with the RUBP (Ribulose-1, 5 bisphosphate) carboxylase enzyme (Koch 1998). These plants grow and compete best in geographical areas where average annual temperatures are below 25°C (Speilmann et al. 1990). About 95% of the Earth’s plants are C$_3$ plants, including most of the grasses and trees in North America (DeNiro and Epstein 1978). The mean $\delta^{13}$C value of C$_3$ plants is -27‰ and ranges between -20‰ and -35‰ (Schoeninger and DeNiro 1984; O’Leary 1988). Tooth enamel tissue representing a C$_3$-only diet would thus have a $\delta^{13}$C value of approximately -13‰ (-27‰ + 11‰ to 14‰).

The C$_4$ photosynthetic pathway, also known as the Hatch-Slack pathway, is the second most common type of photosynthetic pathway used by plants (Speilmann et al. 1990). Plants using the C$_4$ photosynthetic pathway initially fix CO$_2$ by PEP (phosphoenolpyruvate) carboxylase to a four-carbon acid before using the RUBP fixation method (Koch 1998). C$_4$
plants grow best where temperatures are stable, with a slight increase in temperature during the growing season. Many grasses and trees of temperate and tropical regions are C₄ plants, including species such as maize and sugar cane (DeNiro and Epstein 1978). The mean δ¹³C value of C₄ plants is -13‰ and ranges between -17‰ and -9‰ (Schoeninger and DeNiro 1984; O’Leary 1988). Tooth enamel representing a C₄ only diet would generally have a δ¹³C of approximately 1‰ (-13‰ + 11‰ to 14‰).

CAM (Crassulacean Acid Metabolism) photosynthesis is the third and least used form of CO₂ fixation used by plants. Plants which use the CAM photosynthesis pathway may use either C₃ or C₄ photosynthetic pathway (Koch, 1998). All plants which use the CAM pathway are succulents, meaning that they are plants which live in arid regions where drought conditions occur (Speilmann et al. 1990). CAM plants such as cacti and pineapple have a δ¹³C value between -10‰ and -35‰ (Schoeninger and DeNiro 1984; O’Leary 1988).

2.8 Bison Foraging and Herd Behaviour

At the time of European contact, Plains bison flourished over much of the Great Plains of North America with an estimated population of 30 to 50 million individuals (Brink 2008). With a strong European market for bison commodities along with federally supported hunts in the United States, the Plains bison population declined to near extinction (Isenburg 2000). Although conservation over the past century has lifted the population of the bison to sustainable levels and out of the threatened status, the true observation study of “free-ranging” bison has been lost forever. For this reason, the study of extant bison herds along with historic data must be utilized to increase our understanding of behavioral patterns of prehistoric bison populations.

Numerous studies (e.g. Vinton et al. 1993; Fortin et al. 2002; Fortin and Fortin 2009) have been conducted on the foraging behaviour of modern bison herds. The bison, much like the domestic cow (Bos primigenius), use their tongues to pull grass into their mouth. This eating practice alters the species specific selection of foliage being consumed by bison and, as such, they are regarded as unselective grazers (Chisholm et al. 1986; Tieszen 1991). Observational studies (Bamforth 1988; Fortin et al. 2002; Fortin and Fortin 2009) conducted on extant bison herds, suggest bison will generally consume foliage which has the maximum nutrient and energy values obtainable in a short period of time. In other words, bison will choose grazing patches
which are dominated by the grass species with the highest nutrient values during any particular season.

Various other studies (Vinton et al. 1993; Fortin et al. 2002; Fortin and Fortin 2009) of extant bison herds have also provided evidence on the complexity of bison foraging behavior. These observational studies suggest bison often choose to graze on foraging patches which have recently been burned. Fires on grassland biome can remove dead plant materials which increase new growth productivity and perhaps remove invasive species which compete with local grasses (Vinton et al. 1993). Because fires were frequent on the plains prior to European contact (Vinton et al. 1993) bison foraging behaviour would have been influenced by this common occurrence.

Social behavior among extant bison herds has also been observed to dictate their foraging behaviour. Coppedge and Shaw (1998) evaluated particular grazing patterns of sexually segregated bison herds in the Osage Hills of Oklahoma. Their evidence suggested that bison herds of adults bulls (>5 age in yrs) preferred to graze on unburned foraging patches whereas mixed herds (cows, calves, yearlings and young bulls) tended to preferentially graze burned areas. A possible explanation for the different foraging patterns utilized by the two bison social groups may be linked to different reproductive strategies (Coppedge and Shaw 1998). Females may choose locations best suited to obtain fast nutrition values while rearing offspring and bull herds may choose foraging areas best suited for long term energy gain prior to the rut.

Predation risk may also be a contributing factor that influences the foraging behavior of bison. Fortin and Fortin (2009) conducted a study of the foraging behaviors of bison herds located in the Prince Albert National Park. Results of this studied indicated that during the winter months bison foraging behavior was less selective (decrease in plant selection e.g. C. antherodes) due to increased risk of predators (i.e. wolves). Put another way, bison are less selective in foraging patches when predation risk is detected and therefore consume less desirable plant species due to increased time spent scanning for predators (Fortin and Fortin 2009). These authors also noted wolf attacks are more prominent in the winter due to fact the bison herds are smaller during the winter months.

All studies of bison foraging behavior as previously discussed have been conducted on extant bison herds, which face various herd limitations. Some limitations may include park
boundaries or fences on agricultural land and small humanly managed herd sizes when compared to large herds which once existed. Bison skeletal remains in the palaeontological and archaeological record provide evidence that the bison underwent extensive biological changes during the Pleistocene and into the Holocene (Wilson 1978; Bamforth 1988). Because archaeological evidence suggests that the pre-contact peoples of North America hunted bison from the end of the Pleistocene right up until European contact, biological transformations of the bison that affected this animal’s foraging and herd behaviour also may have those who hunted it (Geist 1996). Like every evolutionary lineage of an animal species, the biological history of the modern bison is probably a variation of genetic mixing and localized evolution produced by external conditions (Guthrie 1970; Wilson 1978). A detailed discussion on the evolutionary history of the modern bison is beyond the scope of this paper (see Guthrie 1966 and 1970; Wilson 1978; McDonald 1981; Bamforth 1988; Geist 1996 for detailed discussion). The following is a brief overview of the successive bison species generally thought to have occupied North America during the Pleistocene and into the Holocene.

The first bison species to occupy North America was *Bison priscus*, which is believed to have crossed the Bering Land Bridge from Siberia into Alaska by about 300,000 BP (Wilson 1978; Geist 1996). *B. priscus* perhaps entered a new ecological niche which included carnivore predators such as the saber-tooth cat and flat-faced bear. Under greater predation stress, *B. priscus* evolved into a larger form known as *Bison latifrons* (Geist 1996). *B. latifrons* occupied North America until around 22,000 BP when it was replaced by a smaller-bodied species known as *B. antiquus*. With the end of the Pleistocene Ice Age and the extinction of most megafauna occurring between 10,000 and 11,000 BP, the North American bison began to fill the ecological niche of the developing grasslands in the south (Geist 1996). The earliest form of bison that people hunted in North America is believed to be *B. antiquus*. This species had a larger hump and greater horn span than the modern bison and occupied the southern portion of the Great Plains. *B. antiquus* was hunted by Palaeoindian cultural groups; most notably those of the Folsom cultures (Geist 1996). Folsom kill sites typically include the skeletal remains of one or two adult bison (Geist 1996). Geist (1996) suggests that the common occurrence of one to two bison specimens in Folsom kills may represent adult *B. antiquus* confronting the Folsom hunters in the practice of protecting their young, a protective strategy used by modern muskoxen (*Ovibos moschatus*). Once Folsom hunters understood this protective behaviour of *B. antiquus*, they
could confront a juvenile with the intention of drawing in adult bison within striking distance of a thrown spear.

At around 11,000 BP a smaller-bodied bison species, *Bison occidentalis* crossed Beringia, and migrated into the Plains region of North America (Geist 1996). Perhaps familiar with human interaction in Siberia, *B. occidentalis* lost its confrontation method of defense and developed the defense of flight (Geist 1996). By about 10,000 BP, North America temperatures began to rise and *B. antiquus* moved north where it encountered the ecological niche of *B. occidentalis* (Geist 1996). It has been suggested (Geist 1996) that these two bison species may have interbred and produced a hybrid species of bison, *Bison bison*. With an ongoing warming trend, the grasslands on the North America plains would have flourished, allowing a larger carrying capacity of the grass species consumed by the bison. Benefitting from the expansion of the grasslands, bison populations grew, and their foraging and herd behaviour became more gregarious (Geist 1996). The bison continued to shrink in size, and by about 5000 BP the body type of the bison reached its current form (Geist 1996). Several anatomical changes including: the lengthening of the neck, placement of the eye orbitals on the skull into a more lateral position, and a decrease in limb length of the modern bison when compared to the older species, most likely increased its ability to graze the grasslands of North America (McDonald 1981).

2.9 Brief Overview of the Modern Physical Environments of the Study Area (Southern Saskatchewan)

2.9.1 Climatology

Due to its northern latitudinal position, mid-continental location and its distance from any large water mass, the southern Saskatchewan climate is predominantly controlled by large air mass systems of the atmosphere. In the Koppen climate classification system southern Saskatchewan is characterized as Humid Continental (*Dfb*), with the exception of the south west corner of the province which is classified as a Mid-Latitude Steppe (*BSk*) (Fung 1999). The yearly average temperature range for this region is 44°C but can reach extreme variances of 65°C in any given year (Fung 1999). Winters are generally long and cold and are influenced by the Arctic ridge of high pressure from the north while, summers are cool and short and are influenced by tropical air masses from the south (Fung 1999). In the Saskatoon area, the focus
area of this study, precipitation levels average 300 mm a year with the majority (70%) of it occurring as rain fall during the growing season (May to September inclusive) (Fung 1999). Significant amounts of sunshine (2000+hrs) in a year, as well as frequent high wind or surface pressure values, produce high levels of evapotranspiration (PET) in southern Saskatchewan (Fung 1999). The ecology of Southern Saskatchewan is dictated by this arid climate and, as such, supports a grassland environment.

2.9.2 Terrestrial Ecology

On a broad scale, the natural vegetation of southern Saskatchewan maybe categorized into four Ecoregions distributed into east-west horizontal bands that from north to south comprise the, Aspen Parkland, Moist Mixed Grassland, Mixed Grassland and Cypress Hills, respectively (Acton et al. 1998) Figure 2.1). The area examined in this thesis starts at the Aspen Parkland Ecoregion which includes bluffs of deciduous trees associated with tall fescue grasses. The most common of these grass species is plains rough fescue (Festuca hallii), with a lesser quantities of western porcupine grass (Stipa curtiseta) and June grass (Koeleria macrantha) (Acton et al. 1998). These grass species are highly productive for livestock grazing but are sensitive to over-grazing.

The Moist Mixed Grassland Ecoregion is marked by a decrease in fescue grasses and an increase in mid (20 to 30 cm) and short (<10 cm) grass varieties (Acton et al. 1998). The most common mid-grass species in this zone are western porcupine grass (Stipa curtiseta), needle-and-thread (Stipa comate), green needle grass (Stipa viridula), northern wheat grass (Agropyron dasystachyum), and western wheat grass (Agropyron smithii). Blue grama (Bouteloua gracilis) is the predominant short-grass species (Acton et al. 1998). The Mixed Grassland Ecoregion is predominantly comprised of needle-and-thread and blue-grama grass species. In the driest portions of this Ecoregion, blue-grama grass is the dominant species (Acton et al. 1998). An important note regarding the Moist Mixed Grassland and Mixed Grassland Ecoregions is that under heavy cattle grazing conditions, mid-grass species are often preferably consumed because of their long leaf foliage (Acton et al. 1998). This pattern of discrimination in livestock grazing patterns thus allows short-grass species to flourish after livestock is moved. For this reason, after several years of overgrazing mixed prairie grassland will become dominated by short-grass
Figure 2.1 Map of the Ecoregions of Saskatchewan

Map adapted from Acton et al. 1998
species. The Cypress Hills Ecoregion is marked by an increase in elevation that causes cooler temperatures and increasing precipitation amounts. This Ecoregion includes a mix of fescue grasses which appear on the lower slopes and plateaus of the hills with deciduous trees dominating around mid-slope areas and coniferous species dominating upper slope areas (Acton et al. 1998).

The dominant factor influencing the vegetation in each Ecozone, is the changing of moisture availability mean annual temperatures (Boutton et al. 1980; Laurenroth et al. 1999) from south (drier) to north (wetter). The increase of elevation is the main contributor to the unusual Cypress Hills vegetation. Lower grassland latitude regions tend to be more susceptible to droughts than high latitude treed areas. For this reason, climatic shifts which occur over a significant period of time can alter the ratio of drought tolerant species to moisture-demanding species of grassland areas and even move the vegetation boundaries of an entire ecosystem (Acton et al. 1998). Studies in southern Saskatchewan which have been conducted on plant remains (e.g. Sauchyn 1997; Yansa and Basinger 1999) have provided evidence that vegetation cover in the province has not been static, reflecting fluctuating precipitation and temperature levels throughout the Holocene.

As previously discussed, grasses on the Plains fix carbon in one of two ways. The Calvin Cycle pathway (C\textsubscript{3}) tends to be the mechanism used by the cold tolerant grass species which dominate throughout much of the year; the Hatch-Slack pathway (C\textsubscript{4}) is the photosynthetic pathway used by more drought tolerant grasses which typically prosper late summer into fall. For this reason, the C\textsubscript{3}-to-C\textsubscript{4} percentage ratio of grasses on the Plains is predominantly caused by seasonal change. Because bison consume both C\textsubscript{3} and C\textsubscript{4} grasses, their percentage of dietary intake of C\textsubscript{3} versus C\textsubscript{4} species should change throughout the year. To verify this theory, a study (Tieszen 1994) using isotopic values was conducted on the feces of a grazing bison herd in South Dakota. Results indicated a 45\% increase in consumed C\textsubscript{4} grasses in the late summer. For this reason, carbon isotope values obtained from bison tooth enamel can be used to indicate seasonal variations in the ratio of consumed cold tolerant grass species to warmth tolerant grass species during the amelogenesis of a specific animal.

2.10 Evaluating Dietary and Environmental History Obtained from Isotopic Analysis of Bison Enamel in Northern Great Plains Settings
As previously discussed, mammalian teeth continuously grow over several years and, as such, different types of teeth (e.g. M₁, M₂ and M₃) from a single individual will represent time-specific isotopic data. Bison, generally speaking, have a constrained reproductive cycle where birthing season occurs in spring (Gadbury et al. 2000). For this reason, it can be assumed that the same tooth from any individual will represent the same seasonal period of isotopic data. Further, because teeth form from the crown to the root, precipitated enamel will represent discrete time periods during amelogenesis. The spatial resolution of stable carbon and oxygen isotope varieties in the enamel of any given individual has allowed new applications, including studies of short-term seasonal changes in diet and climate (Fricke et al. 1998; Balasse 2002).

2.10.1 Tracking Climatology and Seasonality using Stable Oxygen Isotopic Compositions

In high and mid-latitudes in North America, temperature changes are drastic from season to season and as such, δ¹⁸O values of precipitation highly correlate with surface temperatures (Dansgaard 1964; Rozanski et al. 1993). On a local scale, δ¹⁸O values may fluctuate for several reasons including the source of precipitation, which is generally seasonally dependant (Amundson et al. 1996). Less positive δ¹⁸O values (-25‰) in the winter to high δ¹⁸O values (-5‰) in the summer occur for several reasons (Fox and Fisher 2001; Passey et al. 2005b). First, seasonal temperatures influence the amount of moisture received by a specific precipitation source (Dansgaard 1964; Rozanski et al. 1993). In the summer, precipitation levels are typically the highest in mid-and-high latitudes, followed by spring, fall and winter. Secondly, evaporation rates are highly affected by temperature change (Dansgaard 1964; Rozanski et al. 1993). Cooler seasons such as spring and fall have lower evaporation levels than summer does, with minimal amounts of evaporation occurring in the winter. Thirdly, moisture sources and changes in storm trajectories also influence δ¹⁸O values (Dansgaard 1964; Rozanski et al. 1993). Summer convection storms precipitate rains that are δ¹⁸O enriched when compared to spring rain values. Fall precipitation typically has δ¹⁸O values between those that occur in summer and spring. Winter precipitation typically occurs in the form of snow or super-cooled rain which characteristically has low δ¹⁸O values. Because bison body water is predominantly composed of ingested drinking water (Gadbury et al. 2000), oxygen isotope analysis on enamel has the capability of inferring climate and seasons during tooth formation.
2.10.2 Tracking Ecology and Seasonality using Stable Carbon Isotopic Compositions

The Northern Great Plains ecosystem supports a mixture of C\textsubscript{3} and C\textsubscript{4} grasses (Sims et al. 1978; French 1979; Boutton et al. 1980; Tieszen 1994; Gadbury et al. 2000). Temperature and precipitation levels highly influence the type of photosynthesis pathway a plant uses (Speilmann et al. 1990). C\textsubscript{3} plants (δ\textsuperscript{13}C value of -27‰), have the advantage where moderate amounts of precipitation is available and low light intensity exists, whereas C\textsubscript{4} plants (δ\textsuperscript{13}C value of -13‰) have the advantage where precipitation is minimal and light intensity is high (Speilmann et al. 1990). During early spring and late fall, when precipitation levels are the highest on the Plains, C\textsubscript{3} plants are most abundant, whereas mid-summer conditions of low precipitation levels and minimal temperatures produce an abundance of C\textsubscript{4} species (Tieszen 1994; Gadbury et al. 2000). Because bison are indiscriminate grazers (Peden et al. 1974; Gadbury et al. 2000) the δ\textsuperscript{13}C value obtained from the carbonate of tooth enamel has the capability of informing on vegetation conditions and seasonality during amelogenesis.
CHAPTER THREE: IMPLICATIONS OF CONDUCTING ANCIENT ENVIRONMENTAL RESEARCH THROUGH THE USE OF STABLE ISOTOPE ($\delta^{18}$O, $\delta^{13}$C) ANALYSES UTILIZING PREHISTORIC BISON TOOTH ENAMEL CARBONATE ON THE NORTHERN GREAT PLAINS

Abstract

The North American Great Plains archaeological record provides evidence that bison ($Bison bison$) skeletal remains are often directly associated with cultural material of ancient human groups. Because of this correlation, archaeologists have increased the importance of scientific research on bison skeletal remains, including isotopic analysis. In this study, bison tooth enamel carbonate from eight archaeological sites, dating to various time periods (~1450-10,350BP) of the Holocene, were isotopically analyzed using stable oxygen isotope ($\delta^{18}$O) and stable carbon isotope ($\delta^{13}$C) ratios for the purpose of inferring ancient seasonality, palaeoclimatic and palaeodietary reconstruction around Saskatoon, Saskatchewan Canada. The isotopic ($\delta^{18}$O & $\delta^{13}$C) results from this study were also measured against isotopic ratios of stable hydrogen isotopes ($\delta$D) and stable carbon isotopes ($\delta^{13}$C) of fossil bison bone collagen from the same eight archaeological sites previously analyzed (Leyden 2004). Finally, stable isotope results from this study ($\delta^{18}$O$_{enamel}$ & $\delta^{13}$C$_{enamel}$) and Leyden (2004) ($\delta$D$_{collagen}$ & $\delta^{13}$C$_{collagen}$) were measured against several previously conducted aquatic proxy studies in the study area to determine if similar palaeoenvironmental conditions are denoted. Fluctuating serial stable oxygen and carbon isotope samples indicate the pattern of seasonal change. Comparisons of the isotopic values of enamel ($\delta^{18}$O and $\delta^{13}$C) and collagen ($\delta$D and $\delta^{13}$C) from bison skeletal remains from the same archaeological sites indicate comparable palaeoenvironmental conditions. Finally, a broad agreement of palaeoenvironmental conditions from various periods of the Holocene in southern Saskatchewan was indicated by both terrestrial and aquatic based proxies.

3.1 Introduction

Palaeoenvironmental studies using various proxies (e.g. Teller and Last 1990; Vance et al. 1997; Yansa 1998; Laird et al. 2007) indicate that climatic conditions and terrestrial vegetation ecology on the northern Great Plains has gone through several transitions during the
Holocene. Although vital to our understanding, the majority of long term palaeoenvironmental studies conducted on the Canadian Plains involve proxies obtained from aquatic settings (i.e. lake cores). Studies have shown that aquatic proxies used to reconstruct surrounding terrestrial environments are not always directly correlated (Hooker et al. 1995). For this reason, a multidisciplinary (aquatic and terrestrial proxies) approach to the development of palaeoenvironmental reconstruction of a particular area should be utilized.

Experimental studies (e.g. Balasse 2002; Passey et al. 2005; Zazzo et al. 2005) as well as studies conducted on fossil bovid biological tissues (e.g. tooth enamel, collagen, dentine) (e.g. Tieszen 1994; Connin et al. 1998; Jahren et al. 1998; Gadbury et al. 2000; Lovvorn et al. 2001; Leyden 2004; Hoppe 2006; Feranec 2007; Higgins and MacFadden 2009; Widga et al. 2010) have provided evidence that carbon isotopic compositions reflect the $^{13}$C/$^{12}$C ratio of an individual’s diet and; therefore, has the potential to indicate the general amount of C$_3$ and C$_4$ grasses consumed during life. Local topography along with the seasonal variations of temperature change and precipitation levels that currently exist on the northern Great Plains, support a mixture of C$_3$ and C$_4$ grasses (Boutton et al. 1980; Tieszen 1994).

Similarly, studies conducted on modern (e.g. Fricke et al. 1998; Hoppe 2006) and fossil (e.g. Higgins and MacFadden 2004; Britton et al. 2009; Bernard et al. 2009; Widga et al. 2010) bovids have indicated that the $\delta^{18}$O isotopic composition of tooth enamel, predominantly reflects the $\delta^{18}$O ratios of drinking water (liquid and water in dietary grass) consumed during an animal’s life. It is also generally understood that oxygen isotopic compositions of meteoric and surface water (water available for terrestrial animal drinking) largely correlates with mean annual surface temperature (i.e. climate) (Dansgaard 1964; Rozanski et al. 1993; Koch 1998). Thus, $\delta^{18}$O isotopic values of tooth enamel carbonate of water dependent animals have the potential to reconstruct ancient seasonality and climates in continental settings.

Numerous studies have applied isotope analysis to various types of bison skeletal remains including bone collagen (Tieszen 1994; Brooks-Lovvorn et al. 2001; Leyden 2004) and tooth enamel (Feranec and MacFadden 2000; Gadbury et al. 2000; Higgins and MacFadden 2004, 2009; Hoppe 2006; Bernard et al. 2009; Widga et al. 2010) for the purpose of indicating shifts in seasonality, climate and plant ecology over particular periods of time and specific continental locations. The goal of this study is to investigate the potential uses of stable oxygen and carbon
isotope ratios from serial sampled bison tooth enamel carbonate, for the purpose of reconstructing ancient seasonality and palaeoenvironmental (climate and plant ecology) conditions on the northern Great Plains during the Holocene. In doing so, scrutiny of mean stable isotope compositions of tooth enamel carbonate ($\delta^{18}O$ and $\delta^{13}C$) (this study) and bone collagen ($\deltaD$ and $\delta^{13}C$), from a previously conducted study (Leyden 2004), is done to determine if similar palaeoenvironmental conditions are indicated by both biological tissues (Figure 2.1). This comparison is validated by using fossil bison remains from the same eight prehistoric archaeological sites that were utilized in Leyden (2004). Finally, exploration of palaeoenvironmental patterns of the Saskatoon area during the Holocene will be conducted by comparing terrestrial and aquatic proxies.

### 3.1.2 Principles of oxygen isotopes used in climate and seasonal forecasting

In the most elementary explanation, oxygen isotope ratios analyzed from both meteoric and surface waters in terrestrial environments will correlate with mean surface temperatures (Dansgaard 1964; Rozanski et al. 1993; Frick and O’Neil, 1996; Higgins and MacFadden 2009). Put another way, warmer weather and/or climate trends will produce higher $\delta^{18}O$ values whereas colder weather/climate trends will produce lower $\delta^{18}O$ values. In mid-to-high latitude regions (45° to 60°) of North America, temperature changes are drastic from season to season and as such, $\delta^{18}O$ values will fluctuate within a seasonal cycle. Further, environmental temperatures in this latitudinal range seldom reach above ~20 °C therefore; the “Amount Effect”, a factor understood to decrease $\delta^{18}O$ values if significant amounts of precipitation and/or humidity is present above ~20 °C, will have minimal effects on the isotope ratios of meteoric waters (Rozanski et al. 1993; Higgins and MacFadden 2009).

Due to its northern latitudinal position, central continental location and its distance from any large water mass, the southern Saskatchewan climate is predominantly controlled by large air mass systems of the atmosphere. The contemporary temperature range in the study area in a general seasonal cycle is large (±40°C), with a mean temperature of -16.7°C in January and 18.4°C in July (Acton et al. 1998). For this reason, $\delta^{18}O$ values should have predictable variations during a specific season, thus creating a sinusoidal curve over the duration of a year. Equally, a sinusoidal curve of $\delta^{18}O$ mean ratios over a long period of time would represent changing climatic trends.
Figure 3.1 Map of study area including the location of archaeological sites used in this study
3.1.3 Carbon isotopes and vegetation forecasts in palaeoenvironmental studies

Mean annual temperature and precipitation levels highly influence the type of photosynthesis pathway a plant uses (Speilmann et al. 1990). The study area is located in the Moist Mixed Grassland Ecoregion of Saskatchewan which predominantly supports a C\textsubscript{3} plant taxa with few C\textsubscript{4} grass species present (Sims et al. 1978; French 1979; Boutton et al. 1980; Tieszen 1994; Gadbury et al. 2000) (Figure 3.2)). C\textsubscript{3} plants which photosynthesize carbon using the Calvin Cycle, are less sensitive to cooler temperatures and have the advantage where moderate amounts of precipitation is available (DeNiro and Epstein 1978). The $\delta^{13}$C value of C\textsubscript{3} plants range between -20‰ and -35‰ with a mean of -27‰ (Schoeninger and DeNiro 1984; O’Leary 1988). C\textsubscript{4} plants which photosynthesize carbon using the Hatch-Slack pathway grow best in regions where temperatures are stable with a slight increase in temperature during the growing season (DeNiro and Epstein 1978). The $\delta^{13}$C value of C\textsubscript{4} plants ranges between -17‰ and -9‰ with a mean of -13‰ and (Schoeninger and DeNiro 1984; O’Leary 1988). On the northern Great Plains, a correlation has been established between minimum temperatures and the relative abundance of C\textsubscript{4} grasses during the growing season (Gadbury et al. 2000).

3.1.4 Bison tooth ontogeny

During amelogenesis (tooth enamel formation), molecular elements (e.g. oxygen, carbon) are present in the lain matrix (dissolved bicarbonates) of secreted enamel (Hillson 1986; Aiello and Dean 1990). Carbon and oxygen isotopes incorporated in the enamel are accumulated from indigested food and water consumed during amelogenesis (Rozanski et al. 1993; Fricke and O’Neal 1996; Higgins and MacFadden 2004). As previously discussed, carbon and oxygen isotopes are source specific and are symbolic of an animal’s physical environment. Once tooth enamel is secreted, it undergoes maturation which includes the deposit of inorganic (biogenic apatite) materials (Suga et al. 1982; Hillson 1986). After maturation occurs, oxygen and carbon isotopes within the enamel do not remodel, and therefore continue to represent the original dietary signals for the remainder of an animal’s life, after death and into the fossil record (Higgins and MacFadden 2009).

Bison have hypsodont (high-crowned) teeth which continuously form over several years (Frison and Reher 1970). Tooth formation occurs in a top-down sequence, enamel secreted
Figure 3.2 Map of the study area and the Moist Grassland Ecoregion of Saskatchewan

Map adapted from Acton et al. 1998
initially forms the crown followed by the cervix (neck) and finally the root (Hillson 1992). The third mandibular molar (M₃) (tooth used in this study) of a bison generally begins forming at 9 months and continues until the animal is about ca. 2 years of age (Gadbury et al. 2000). Experimental studies (e.g. Ambrose and Norr 1993; Tieszen and Fagre 1993; Howland et al. 2003; Jim et al. 2004; Kellner and Schoeninger 2007; Warinner and Tuross 2009) have indicated that isotope values obtained from enamel carbonate reflect an animal’s integrated (overall) diet but at a fractionated value. Carbon isotope values from tooth enamel of large (>100 kg) ungulates have been measured to have a fractionated value of +14.6‰ compared to its actual diet (Passey et al. 2005b).

Carbon and oxygen isotopic ratios analyzed from a bison M₃ may be considered to be the best tooth for palaeoenvironmental research for several reasons. First, because this tooth is formed post-weaning, isotopic ratios from this tooth will not contain potential aberrant results caused by nursing or en utero (biological synthesis) effects (Fricke and O’Neil 1996; Gadbury et al. 2000; Balasse 2002). Secondly, due to the sequence ontogeny of tooth enamel, sequential subsamples of M₃ enamel should produce a time-series of isotope ratios representative of dietary and environmental changes over an approximate 15 to 18 month period (Fricke and O’Neal 1996).

3.2 Materials

All fossil bison teeth utilized in this study were collected from archival collections housed in various locations within the province of Saskatchewan. The criteria used for the archaeological site selection as set forth by Leyden (2004) was based on several factors including but not limited to; the close proximity of archaeological sites to one another; and the time range this sample collection represents. In total, eight archaeological sites were chosen, seven of which are located within a 50 km radius to the city of Saskatoon, with the eighth site situated approximately 200 km southwest of Saskatoon (Figure 3.1). Accelerator mass spectrometry (AMS) dates have been obtained from all of the archaeological sites used in this study with the exception of the Norby and Tschetter sites, which have been dated using an averaged value of three radiometric dates (Table 3.1). Based on time-diagnostic cultural material in the form of projectile points associated with the excavated bison remains, six human cultural affiliations are
Table 3.1 Archaeological sites used in this study

<table>
<thead>
<tr>
<th>Site</th>
<th>Borden Designation</th>
<th>Cultural Association</th>
<th>Cal yr BP</th>
<th>14C yr. BP</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heron Eden (HE)</td>
<td>EeOi - 11</td>
<td>Cody</td>
<td>10,352 ± 50</td>
<td></td>
</tr>
<tr>
<td>Gowen (GW)</td>
<td>FaNq - 32</td>
<td>Mummy Cave</td>
<td>6,653 ± 103</td>
<td></td>
</tr>
<tr>
<td>Norby (NY)</td>
<td>FaNq - 56</td>
<td>Mummy Cave</td>
<td>5,808 ± 33*</td>
<td></td>
</tr>
<tr>
<td>Amisk (AM)</td>
<td>FbNp - 17</td>
<td>Oxbow (Level 4)</td>
<td>4,941 ± 103</td>
<td></td>
</tr>
<tr>
<td>Harder (HD)</td>
<td>FbNs - 1</td>
<td>Oxbow</td>
<td>4,823 ± 33</td>
<td></td>
</tr>
<tr>
<td>Thundercloud (TH)</td>
<td>FbNp - 25</td>
<td>McKean (Level 5)</td>
<td>3,804 ± 13</td>
<td></td>
</tr>
<tr>
<td>Fitzgerald (FG)</td>
<td>EiNp - 8</td>
<td>Besant</td>
<td>1,442 ± 101</td>
<td></td>
</tr>
<tr>
<td>Tschetter (TC)</td>
<td>FbNr - 1</td>
<td>Old Woman’s</td>
<td>1,035 ± 40**</td>
<td></td>
</tr>
</tbody>
</table>

Cultural associations have been determined, based on projectile point associations, by independent and previously conducted studies (see Corbiel 1995; Walker; 1992; Zurburg 1991; Amundson 1986; Dyck 1977; Webster 1999; Hjermstad 1996; Linnamae 1988). For multi-component sites (Amisk and Thundercloud) the levels have been indicated. Bone samples used in this study are only taking from these excavation levels. Cal yr BP (calibrated radio carbon years before present) 14Cal yr. BP (conventional radio carbon years before present) All Cal yr BP dates have been determined by Leyden (2004). *Averaged conventional radio carbon date determined by Zurburg (1991). Λ The Norby site has also been designated with a ~7,800 year old date (see Appendix A of this paper for details). ** Averaged conventional radio carbon date determined by Linnima (1988).

represented and span an approximate 9000-year time period of human occupation within the study area.

Eight well preserved fully developed third bison mandibular molars (M₃) with a minimal crown length of 50 mm were used to represent each of the eight prehistoric archaeological sites utilized in this study. Though it would have been ideal to have numerous fully developed M₃ bison molars represent each particular bison population, prehistoric archaeological (e.g. camp and occupational) sites on the northern Great Plains often do not include large quantities of bison teeth in cultural levels. Suffice to say that other than the tongue, the contents of the head or skull of the bison is not regarded as a high nutritional valued item; therefore bison teeth are sparse in prehistoric camp or occupational sites on the northern Great Plains. The author of this thesis is
not aware of any statistical data which exists on the percentage of bison kill/butchery sites versus camp/occupational sites on the northern Great Plains. Based on personal field archaeological experience, camp/occupational sites far outnumber kill/butchery sites on the northern Great Plains and therefore fully mature unworn third bison mandibular molars are rarely found in these types of archaeological sites.

3.3 Methods

The sampling method (Figure 3.3) used in this study is comparable to previous publications (Bryant et al. 1996b; Fricke and O’Neil 1996; Gadbury et al. 2002; Balasse 2002; Bernard et al. 2009; Widga et al. 2010) which have focused on serial sampling of tooth enamel

**Figure 3.3** Schematic of tooth enamel carbonate sampling protocol used in this study

of hypsodont dentition. It is worth noting, that Zazzo et al. (2005) provided evidence, through a microsampling technique of tooth enamel, that the sampling strategy used in this study will result in some degree of isotopic ratio dampening based on the fact that tooth enamel mineralization
occurs not only root to crown but also from the cementum-enamel junction (CEJ). This study uses teeth from the same species and therefore; isotopic ratios of each sampled tooth should have the same degree of isotopic mixing, consequently providing accurate information for the purpose of reconstructing environmental conditions which existed during a particular animal’s life.

Prior to enamel collection, each tooth was manually cleaned with stainless steel dental tools followed by a light buffering with a Dremel™ tool with a sanding drum attachment in order to remove any calculus or foreign material adhering to the enamel surface. Each tooth was then washed (3X) with distilled water and set aside to dry. Using a Dremel™ with a diamond drill bit, enamel powder samples were collected from the metaconid cusp of the tooth in approximately 3mm bands perpendicular to the growth axis and through the entire thickness of the enamel stopping at the CEJ. Eight samples were collected over the entire length of the tooth (crown to root) in approximately 5-6 mm intervals, with the first sample collected from the crown tip (oldest) and sample 8 (youngest) collected from the enamel closest to the root.

Powdered enamel was roasted in a vacuum oven at 200°C for 1 hour to remove water and volatile organic contaminants that may confound stable isotopes values of carbonates. Using a Finnigan Kiel-IV carbonate preparation device, approximately 50 micrograms of carbonate was reacted at 70°C with 3 drops of anhydrous phosphoric acid for 420 seconds. The CO₂ evolved is then cryogenically purified before being directly passed on to the dual inlet of a Finnigan MAT 253 isotope ratio mass spectrometer in the Saskatchewan Isotope Laboratory, located at the University of Saskatchewan. Isotope ratios are corrected for acid fractionation and ¹⁷O contribution using the Craig correction (Coplen 1994) and reported using standard delta (δ) and in per mill (‰) notations relative to V-PDB scale. Data is directly calibrated against the international standard NBS-19 that is by definition δ¹³C = 1.95‰ and δ¹⁸O = -2.2‰ V-PDB. Accuracy of δ¹³C and δ¹⁸O are 0.0 and 0.11‰, respectively based on (n=25) carbonate samples of tooth enamel. δ¹⁸O samples are reported in V-SMOW using the Coplen (1994) equation where;

\[ \delta^{18}O_{\text{V-SMOW}} = 1.03092 \times \delta^{18}O_{\text{V-PDB}} + 30.92. \]

3.4 Results

3.4.1 Bison Teeth
Enamel carbonate from eight fully developed adult bison molars (M3) from eight different prehistoric archaeological sites were sequentially sampled from apex to the cervix for δ18O and δ13C isotopic analysis. A total of 64 samples were collected, eight sub-samples from each tooth were compared for the purpose of indicating seasonality (Table 3.2). In addition, the mean value of δ18O and δ13C sub-samples from each tooth was calculated for the purpose of comparing and contrasting climatic and vegetation conditions of the Saskatoon, Saskatchewan area during various time periods of the Holocene. The results of δ18O and δ13C isotopic ratios from this study are presented below by each individual archaeological site.

**Table 3.2 Oxygen and carbon isotope results of bison (M3) sequential samples**

<table>
<thead>
<tr>
<th>Site</th>
<th>Sample</th>
<th>δ18O V-SMOW (%)</th>
<th>δ13C V-PDB (%)</th>
<th>Lab ID #</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heron Eden (HE)</td>
<td>1</td>
<td>14.9</td>
<td>-6.70</td>
<td>7690</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>16.4</td>
<td>-8.04</td>
<td>7691</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>17.7</td>
<td>-8.52</td>
<td>7692</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>15.8</td>
<td>-8.42</td>
<td>7693</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>14.7</td>
<td>-7.68</td>
<td>7694</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>14.8</td>
<td>-7.66</td>
<td>7695</td>
</tr>
<tr>
<td></td>
<td>7</td>
<td>15.5</td>
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</tr>
<tr>
<td></td>
<td>8</td>
<td>17.3</td>
<td>-7.50</td>
<td>7697</td>
</tr>
<tr>
<td>Gowen (GW)</td>
<td>1</td>
<td>14.2</td>
<td>-6.70</td>
<td>7074</td>
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<td></td>
<td>2</td>
<td>14.3</td>
<td>-6.5</td>
<td>7073</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>14.8</td>
<td>-6.9</td>
<td>7078</td>
</tr>
<tr>
<td></td>
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3.4.2 Heron Eden site

The Heron Eden (~10,352 BP) site is the oldest archaeological site analyzed in this study, and is the only site outside a 50 km radius of the city of Saskatoon. Sequential sub-samples of δ¹⁸O had a range between 14.7‰ and 17.7‰ and oscillate periodically, a symbolic pattern resembling the change of winter and summer watering (Table 3.2, Figure 3.4). The δ¹³C sub-sample values had a range of -8.5‰ to -6.7‰ and fluctuated longitudinally along the tooth axis, interpreted as seasonal diet change of C₃/C₄ grasses consumed during tooth development. The mean δ¹⁸O and δ¹³C isotope values of tooth enamel carbonate analyzed from the Heron Eden bison specimen were 15.9‰ and -7.8‰, respectively.

Figure 3.4 δ¹⁸O and δ¹³C sub-sample results from the Heron Eden site
3.4.3 Gowen site

The Gowen site specimen dates to the Middle-Holocene (~6653 BP) and also had a sinusoidal curve in both the δ¹⁸O and δ¹³C sub-samples (Table 3.2, Figure 3.5). The range variation of δ¹⁸O values between the summer maxima (18.5‰) and winter minima (14.2‰) of this animal was 4.3‰ over an approximate 15 to 18 month period. The δ¹³C sub-samples had a range of -6.5‰ and -8.0‰ and correlated with the maxima and minima δ¹⁸O subsamples indicating that C₃ grasses were consumed during the peak period of the growing season. The Gowen site specimen had a mean δ¹⁸O value of 16.2‰ and a mean δ¹³C value of -7.3‰.

Figure 3.5 δ¹⁸O and δ¹³C sub-samples results from the Gowen site
3.4.4 Norby site

The Norby site also dates (~5808 BP) within the middle period of the Holocene. The δ¹⁸O value range between the highest (18.3‰) and lowest (11.9‰) sub-samples of this tooth was large at 6.4‰ (Table 3.2, Figure 3.6). The most positive δ¹³C (-7.4‰) sub-sample occurred near the cervix of the tooth whereas the most negative δ¹³C (-8.6‰) subsample was near the crown. The mean δ¹⁸O and δ¹³C values associated with the Norby site specimen are (14.9‰) and (-8.0‰), respectively.

Figure 3.6 δ¹⁸O and δ¹³C sub-samples results from the Norby site
3.4.5 Amisk site

The Amisk site is dated (~4941 BP) to a transitional episode between the middle-and-late time periods of the Holocene. Both the $\delta^{18}O$ and $\delta^{13}C$ sub-samples oscillate down the axis of the tooth creating sinusoidal curves symbolic of seasonal temperature changes (Table 3.2, Figure 3.7). The highest $\delta^{18}O$ (17.9‰) and lowest $\delta^{13}C$ (-7.5‰) sub-samples values of this tooth are associated with the growing season, whereas the depleted $\delta^{18}O$ (13.7‰) and enriched $\delta^{13}C$ (9.0‰) represent the fall/winter season of the sinusoidal curves. The mean $\delta^{18}O$ value was 15.6‰ and the mean $\delta^{13}C$ value was -8.5‰ for the Amisk specimen.

Figure 3.7 $\delta^{18}O$ and $\delta^{13}C$ sub-samples results from the Amisk site
3.4.6 Harder site

Like the Amisk site, the Harder site also dates (~4823 BP) within the transitional time period of the Holocene. The periodicity between the maximum and minimum $\delta^{18}$O sub-samples from this tooth was 18.4‰ to 15.2‰ (Table 3.2, Figure 3.8). Carbon isotope sub-sample values from the Harder site had the largest swing (2.1‰) from -4.8‰ to -6.9‰ of all teeth analyzed in this study. Interpretation of this large swing pattern in $\delta^{13}$C values of this animal may be a result of an increased percentage of a C$_4$ diet compared to all other animals analyzed. The mean oxygen value from the Harder site was 16.96‰ and -5.84‰ carbon.

**Figure 3.8** $\delta^{18}$O and $\delta^{13}$C sub-samples results from the Harder site
3.4.7 Thundercloud site

The Thundercloud site dates (~3804 BP) within the late period of the Holocene. The range between the δ\(^{18}\)O summer maxima (18.2‰) and winter minima (14.9‰) was moderate at 3.3‰ when compared to all other teeth analyzed in this study (Table 3.2, Figure 3.9). The δ\(^{13}\)C sub-sample values of the Thundercloud animal ranged between a maximum of -7.9‰ and a minimum -9.58‰. Mean oxygen and carbon isotope ratios from the Thundercloud site were 16.4‰ and -8.9‰, respectively.

Figure 3.9 δ\(^{18}\)O and δ\(^{13}\)C sub-samples results from the Thundercloud site
3.4.8 Fitzgerald site

The Fitzgerald site dates (~1442 BP) to the late time period of the Holocene. The Fitzgerald tooth had the largest $\delta^{18}$O sub-sample range (22.1‰ to 15.1‰) in this study (Table 3.2, Figure 3.10). Similarly, the value range between the maximum (-5.0‰) and minimum (-6.9‰) $\delta^{13}$C sub-sample values was also large. The mean $\delta^{18}$O (18.3‰) and $\delta^{13}$C (-5.84‰) values of this animal were the most positive values of all teeth analyzed in this study.

**Figure 3.10** $\delta^{18}$O and $\delta^{13}$C sub-samples results from the Fitzgerald site
3.4.9 Tschetter site

The Tschetter site dates (~1035 BP) within the late period of the Holocene and represents the youngest time period in this study. The oxygen isotopic range between 18.9‰ and 12.5‰ sub-sample values was significantly higher than the majority of all analyzed teeth (Table 3.2, Figure 3.11). On the contrary, the carbon sub-samples of the Tschetter tooth had the lowest range between -8.40‰ and -9.20‰ of all teeth analyzed in this study. Similarly, the mean \( \delta^{18}O \) (14.8‰) and \( \delta^{13}C \) (-8.8‰) values of the Tschetter tooth were also the lowest among all other archaeological sites represented in the study.

Figure 3.11 \( \delta^{18}O \) and \( \delta^{13}C \) sub-samples results from the Tschetter site
3.5 Discussion

3.5.1 Meaning of the $\delta^{18}O$ compositions of bison tooth enamel carbonate

All analyzed teeth showed various levels of seasonal variability as a result of fluctuating $\delta^{18}O$ values in the meteoric water, which is typical in the latitudinal range of the study area. The sinusoidal curve created by oscillating $\delta^{18}O$ values along the longitudinal axis of each individual tooth is a strong argument that the original $\delta^{18}O$ signal has been preserved and is symbolic of changing temperatures from winter (low $\delta^{18}O$ values) to summer (high $\delta^{18}O$ values) seasons. Further, the comparison of the mean $\delta^{18}O$ results from the individual teeth analyzed in this study implies that climatic conditions in the study area were not static, but rather fluctuated throughout the Holocene.

The Fitzgerald and Tschetter sites had the largest range between the maxima (summer) and minima (winter) $\delta^{18}O$ sub-sample values in this study. Similarly, the mean $\delta^{18}O$ value results from the Fitzgerald and Tschetter sites also represent the warmest and coolest climatic conditions in this study, respectively. The Norby and Gowen sites also had a large variance between the high and low $\delta^{18}O$ sub-sample values. Mean $\delta^{18}O$ values from the Norby site corresponds to a climatic cooling trend when compared to the Gowen site. The Harder, Thundercloud, Amisk and Heron Eden sites all had moderate variability (4.1‰ to 3.0‰) between the summer maximum and winter minimum $\delta^{18}O$ values. Enriched mean oxygen isotope values are associated with the Harder and Thundercloud tooth samples when compared to Amisk and Heron Eden sites. These mean $\delta^{18}O$ results suggest warmer meteoric water was available to drink for the Harder and Thundercloud animals then the Amisk and Heron Eden specimens.

3.5.2 Meaning of the $\delta^{13}C$ compositions of bison tooth enamel carbonate

All animals analyzed in this study had variations of carbon isotope compositions in tooth samples which are interpreted as reflecting changes in the amount of $C_4$ grasses consumed during tooth development. The Harder, Fitzgerald and Heron Eden animal’s had the largest $\delta^{13}C$ sub-sample range $>1.9‰$, signifying an increase in the relative abundance of $C_4$ versus $C_3$ grasses during tooth development. With the exception of the Tschetter site, all other archaeological sites represented in this study had a maxima and minima $\delta^{13}C$ sub-sample range between 1.7‰ and 1.2‰. The Tschetter animal had the smallest $\delta^{13}C$ sub-sample variation $<1‰$, signifying
minimal amounts of C₄ grasses were consumed during an approximate 18 month period required for tooth formation.

Mean carbon isotope results indicate that the Fitzgerald and Harder animals consumed the largest quantities of C₄ grasses when compare to all other archaeological sites represented in this study. High concentrations of C₄ grasses in the study area may indicate a combination of prolonged periods of high to moderate temperatures along with high aridity levels during the growing season. The Thundercloud, Tschetter and Amisk animals had the lowest mean δ¹³C values, which would suggest minimal amounts of C₄ grasses were consumed during the development of the third mandibular molar. Low mean δ¹³C values in the study area may signify cooler climatic conditions and possibly increased precipitation levels during the growing season. The Norby, Heron Eden and Gowen sites had mean δ¹³C values representing intermediate amounts of C₄ grasses when compared to the other archaeological sites analyzed in this study.

3.5.3 Comparing mean isotope compositions from bison tooth enamel (δ¹³C and δ¹⁸O) and bone collagen (δ¹³C and δD) from the same archaeological sites as palaeoenvironmental proxies.

In a previous study (Leyden 2004), carbon and hydrogen isotope compositions were analyzed from bison bone collagen, for the purpose of indicating palaeoenvironmental conditions, from the same archaeological sites used in this study (Table 3.3). In order to increase the groundwork for isotope applications in the reconstruction of ancient environments, comparisons have been made between the results of this study and Leyden (2004). The purpose of this comparison was threefold; to scrutinize trace isotopic uptake in various biological tissues of the same species from the same archaeological sites and determine if the palaeoenvironmental signals are similar; determine if any discrepancies occur between hydrogen and oxygen isotopes in tracing the mean annual surface temperatures of a specific study area; and finally, determine if isotope analysis (δ¹⁸O, δ¹³C) from a single bison tooth indicates similar palaeoenvironmental conditions at specific periods of time compared to that of isotope analysis (δD, δ¹³C) of bone collagen from multiple specimens.
Table 3.3 Mean isotopic compositions from enamel (δ\(^{18}\)O, δ\(^{13}\)C) and collagen (δD, δ\(^{13}\)C) from bison skeletal remains from the same archaeological sites.

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Similar to tooth enamel, collagen contains various isotopic species (e.g. carbon, hydrogen) which are derived from dietary intake and are also environmentally source specific (Ambrose and Norr 1993). Although collagen has a similar correlation between isotope values and the consumer’s diet, much like tooth enamel, several discrepancies exist. Experimental feeding studies (Ambrose and Norr 1993; Tieszen and Fagre 1993) have indicated that isotope signatures associated with bone collagen only represent the protein portion of an animal’s diet and not the overall diet. Further, collagen isotope compositions continually change throughout the life of an individual animal, this process is often referred to as ‘turn-over’ (Jorkov et al. 2009). For this reason, dietary isotopic inputs obtained from collagen will be time averaged. Although difficult to estimate (see Jorkov et al. 2009 for detail discussion), suggestions have indicated that collagen turnover rate is between 10 and 20 years. Finally, carbon isotope data of bone collagen has an averaged +5.0‰ enrichment when compared to the actual diet (Ambrose and Norr 1993).

Hydrogen (δD) will isotopically fractionate much like oxygen in meteoric precipitations (Ehleringer and Dawson 1992; Gat 1996). The theoretical chemical reaction of hydrogen (δD) in the hydraulic cycle is therefore also temperature-dependent (Gat 1996). For this reason, several studies (e.g. Cormie 1991; Cormie et al. 1994a, Cormie et al. 1994b; Leyden et al. 2004) have
used δD values of modern and fossil mammal tissues to infer temperature regimes at differing continental locations.

Palaeoclimatic conditions verified from stable (δ¹⁸O enamel and δD collagen) isotopic analysis from fossil bison remains from the same archaeological sites are comparable at several archaeological sites and different at others. The Fitzgerald site had the most positive mean δ¹⁸O enamel and δD collagen values when compared to all archaeological sites utilized in this study. As previously discussed, high δ¹⁸O and δD values of meteoric waters are generally reflective of increased temperatures in the study area. For this reason, interpretations may be made that the Fitzgerald bison population occupied warmer climatic conditions than all other bison populations analyzed in this study. Though the isotopic signals from both δ¹⁸O enamel and δD collagen were moderately variable at the Harder, Thundercloud and Tschetter sites, the general trend line in Figure 3.12 is comparable. This suggests that both the δ¹⁸O enamel and δD collagen values from these sites indicate similar warming and cooling patterns in the late-Holocene period. Moderate palaeoclimatic results were indicated by the mean δ¹⁸O enamel and δD collagen values at the Heron Eden and Gowen sites. δD collagen values indicate that a slight cooling trend occurred from the earlier part of the Holocene to the middle period. On the contrary, the mean δ¹⁸O enamel values from the Heron Eden and Gowen sites represents a small warming transition occurred between the early-and-middle portions of the Holocene.

Significant variability occurred between the comparison of the mean δ¹⁸O enamel and δD collagen at the Amisk and Norby sites. Enriched mean δD collagen values were associated with the Norby and Amisk sites, which would signifying warmer meteoric water was available for drinking at these two archaeological sites when compared to the majority of all other bison populations analyzed in this study. Opposing results were indicated by the mean δ¹⁸O enamel values of the Norby and Amisk sites which suggest that cooler temperatures were present during the time periods represented by these two archaeological sites. Reasons for the variability in palaeoclimatic conditions between the mean δD collagen and δ¹⁸O enamel values from the Amisk and
Norby sites is not clear but may include: the duration of time represented by bison enamel (~18 months) and collagen (10-20 years); the possibility that the climatic conditions associated with the middle Holocene period are highly variable; and the possibility that palaeoclimatic signals from hydrogen and oxygen isotopes from various biological tissues is not equivalent. More work is needed to understand this variation.

Mean carbon \((\delta^{13}C)\) isotope values from both bison enamel and collagen correlated relatively well at all eight archaeological sites (Figure 3.13). A notable difference detected between mean \(\delta^{13}C_{\text{collagen}}\) and \(\delta^{13}C_{\text{enamel}}\) values in Figure 3.13 is the exaggerated amplification of the trend line between each archaeological site. The author interprets that the carbon isotope ratios from tooth enamel (integrated diet) are more exaggerated than collagen (protein portion of diet) based on the differences of biological tissues isotopically analyzed and not a result of dietary differences. Like the mean hydrogen and oxygen isotopes indicated, the ancient vegetation ecosystem of the study area has changed throughout the Holocene.
3.5.4 Palaeoenvironmental overview of the study area during the Holocene based on terrestrial and aquatic derived proxies

In Saskatchewan, evidence from numerous proxies including mineralogy and lithology (Teller and Last 1990), palaeobiology (Vance et al 1997), palaeobotany (Yansa 1998), and diatoms (Laird et al 2007) analyzed from lake cores have indicated that several warming and cooling trends occurred since that last glacial event (Figure 3.14). The broad-scale perspective of these palaeoenvironmental proxy studies indicate that the early part of the Holocene is characterized as a cool and moist climatic trend; the middle period of the Holocene as a warm and arid climate episode; and the Late-Holocene characteristic of more moderate climatic conditions with sporadic warming and cooling events. For simplicity, we will discuss the palaeoenvironmental conditions of Saskatchewan in this three time period scale.
Figure 3.14 Multi-proxy climate change panorama of southern Saskatchewan during the Holocene

- A - Kenosee Lake: Mineralogy and palaeobotany proxies (Vance et al. 1997)
- B - Ceylan Lake: Lithology and mineralogy proxies (Teller and Last 1990)
- C - Waldsea Lake: Lithology and mineralogy proxies (Teller and Last 1990)
- D - Oro Lake: Diatoms and mineralogy proxies (Laird et al. 2007)
- E - Andrews Lake: Palaeobotany proxy (Yansa 1988)
- F - Bison Bone Collagen ($\delta^{13}$C) mean values (Leyden 2004)
- G - Bison Tooth Enamel ($\delta^{18}$O) mean values (this study)
3.5.4.1 Early-Holocene (~10,000-7500 cal years BP)

Palaeoenvironmental proxy evidence from Ceylon (Teller and Last 1990) and Oro (Laird et al 2007) lakes in Saskatchewan indicate that Early-Holocene had sufficient precipitation levels able to recharge water body levels. Palaeobotany records from Andrews Lake signify that during the early-Holocene a transition occurred from coniferous (spruce forest) trees to deciduous parkland, which would suggest increased mean annual temperatures to more recent time (Yansa 1998). Similarly, a study (Landi et al 2002) conducted on stable carbon isotope values of pedogenic materials in southern Saskatchewan, indicated that fewer C$_4$ grass species existed in the northern Great Plains during the early-Holocene. The results from Landi et al (2002) would suggest that a gradient increase in mean annual temperatures occurred during the early-Holocene continuing into the mid-Holocene.

The Heron Eden site is the only archaeological site used in this study which dates within the Early-Holocene period. Similar to the findings of the aquatic proxies discussed above, mean isotopic values from bison enamel ($\delta^{18}$O, $\delta^{13}$C) and collagen ($\delta$D, $\delta^{13}$C) from the Heron Eden site indicate a period of increased precipitation levels and moderate to low abundance of C$_4$ grasses.

3.5.4.2 Mid-Holocene (~7500-5000 cal years BP)

The general consent of several aquatic based palaeoenvironmental studies (e.g. Teller and Last 1990; Laird et al. 2007) conducted in southern Saskatchewan, denote that the region went through an arid climatic episode during the mid-Holocene. This warm climatic episode is often referred to as the “Altithermal”, “Mid-Holocene Climatic Optimum” and more recently the “Hypsithermal”. Once thought to be a strictly warm and arid period (Antevs 1948 and 1955), increased development of palaeoenvironmental proxies in the last two decades has established a greater understanding of the Mid-Holocene conditions and has produced data which is highly variable even in localized region. In southern Saskatchewan, diatom and mineralogy records from Oro Lake indicate high temperatures and aridity levels occurred during the first half of the Mid-Holocene and returned to moderate conditions in the last half (Laird et al. 2007). Mineralogy and lithology records from Ceylon and Waldsea Lakes indicate that peak arid conditions occurred during the last (~6500 to 4800 BP) half of the Middle Holocene (Teller and Last 1990).
Both the Gowen and Norby archaeological sites date within the Mid-Holocene. δD values from bison bone collagen from the Norby site represent a warm climatic episode similarly indicated by the lake core studies from Ceylon and Waldsea Lakes. These warm mean annual temperatures are typical of Hypsithermal type conditions. Interestingly, δ¹³C collagen values from the Norby site are moderate when compared to the other archaeological sites measured in this study, indicative of a more temperate climate. Oxygen and carbon stable isotope values from tooth enamel from the Norby site indicate a cooler climatic period with increased moisture levels. The environmental signals represented by the δ¹⁸O and δ¹³C of tooth enamel of the Norby site specimen are similar to those conditions denoted by diatom and mineralogy proxies from Oro Lake, suggesting a period of higher effective moisture (Laird et al 2007).

Stable isotope analysis from both bison collagen (δD and δ¹³C) and enamel (δ¹⁸O and δ¹³C) from the Gowen site suggest moderate mean annual temperatures and a slight increase of C⁴ grass accessibility for bison foraging when compared to all other archaeological sites considered. Similar environmental conditions, which date to the approximate time period of the Gowen site, have been indicated from a multi-proxy lake core study at Oro Lake (Laired et al 2007).

3.5.4.3 Late-Holocene (~5000 cal years BP to present)

The broad perspective of environmental conditions of the Late-Holocene in southern Saskatchewan is generally described as a period marked by an increase of precipitation levels and cooler climes when compared to the mid-Holocene. On the contrary, the narrow perspective of the palaeoenvironmental conditions on the northern Great Plains during the Late-Holocene is variable with sporadic increase in both warm/dry and cool/wet climatic episodes. Sediment core records from Ceylon and Waldsea Lakes indicate a sharp transition occurred during the early portion of the Late-Holocene, from extremely low lake levels associated with the Hypsithermal, to increasingly high lake levels (Teller and Last 1990). Similarly, temperature decreases and a decline in C⁴ grass abundance, a general indication of increase precipitation levels, is also detected by δD collagen, δ¹³C collagen and δ¹³C enamel from the Amisk site which has been dated to the early portion of the Late-Holocene. The Harder site also dates to the early part of the Late-Holocene. Contrary to the Amisk isotope results, mean δ¹⁸O enamel values from the Harder site denote a sharp increase in mean annual temperature which correlates well with the increase in C⁴
grass species denoted by $\delta^{13}C_{\text{enamel}}$ and $\delta^{13}C_{\text{collagen}}$ values from the same archaeological site. Results from a multi-proxy (mineralogy and palaeobiology) study from lake-cores extracted from Kenosee Lake, indicate maximum low lake levels around the same time period as the Harder site (Vance et al. 1997).

Between ~4500 and 1800 BP, intermittent climate conditions have been indicated in several palaeoenvironmental proxies studies in southern Saskatchewan, including cool/wet conditions inferred by sediment cores from Ceylon Lake (Teller and Last 1990) to warm/dry conditions indicated from mineralogy and palaeobiology proxies from Kenosee (Vance et al. 1997) and Oro (Laird et al., 2007) Lakes. Stable isotope values from both bison collagen ($\delta D$ and $\delta^{13}C$ ) and tooth enamel ($\delta^{18}O$ and $\delta^{13}C$) from the Thundercloud site, which has been dated to ~3800 BP, represents a period of cooler and/or wetter climate conditions and a decline in $C_4$ grass species.

Increased aridity levels resulting in low lake levels have been reported from several lake-core studies done in southern Saskatchewan between ~1500 and 800 years BP (Teller and Last 1990; Vance et al. 1997). Enriched mean $\delta^{18}O$ and $\delta^{13}C$ from enamel and $\delta D$ and $\delta^{13}C$ from bone collagen (Leyden 2004) from the Fitzgerald (~1400 BP) bison population signifies extreme mean annual temperatures and high aridity levels when compared to all other archaeology sites analyzed in this study. A short warm climatic episode referred to as the “Medieval Warm Period” has been described in great detail in many studies (see Clague et al. 2010 for detailed discussion) and is generally reported between 1100 and 800 years BP.

Following the Medieval Warm Period, an episode of increased cool/wet conditions has been reported and is referred to as the Little Ice Age (see Clague et al. 2010 for detailed discussion). Numerous (e.g. Teller and Last 1990; Vance et al. 1997; Laird et al., 2007) studies conducted on lake cores in southern Saskatchewan have recorded the Little Ice Age by an abrupt increase in lake levels between ~800 and 100 years BP. Similar, mean $\delta^{18}O$ and $\delta^{13}C$ values from tooth enamel from the Tschetter (~1035 BP) site represent an abrupt increase in moisture levels and a drastic decrease in mean annual temperatures but at an earlier time. Mean $\delta D$ and $\delta^{13}C$ values from bone collagen (Leyden 2004) from the Tschetter site also indicate a cool/wet climatic trend but to a lesser extent.

3.6 Conclusions
For the first time $\delta^{18}$O and $\delta^{13}$C values from bison teeth carbonate have been analyzed for the purpose of indicating short-term (seasonality) and long-term (climate trends) conditions in southern Saskatchewan during the Holocene. The preservation of the original oxygen and carbon stable isotope signals in bison molars was successful in identifying seasonal temperature fluctuations in an approximate 15 to 18 month period. Mean $\delta^{18}$O and $\delta^{13}$C values analyzed from bison teeth from eight different archaeological sites showed significant changes. The variability in mean $\delta^{18}$O and $\delta^{13}$C values at different archaeological sites is interpreted as representing changes in climatic and vegetation conditions over time.

Analysis of mean isotopic values in the enamel ($\delta^{18}$O and $\delta^{13}$C) and collagen ($\delta$D and $\delta^{13}$C) of fossil bison from southern Saskatchewan revealed similarities in the majority (but not all) archaeological sites. Carbon ($\delta^{13}$C) values from both bison enamel and collagen correlated relatively well at all eight time periods represented in this study. This evidence suggests that carbon isotope analysis on both fossil bison skeletal tissues similarly and independently infers that habitat conditions can be predicted based on the abundance of C$_3$/C$_4$ grasses consumed by fossil bison populations.

Some variability occurred in inferring climatic conditions between tooth enamel ($\delta^{18}$O) and bone collagen ($\delta$D) from fossil bison from the same archaeological sites. The moderate and significant climatic differences observed between these two biological tissues at several different archaeological sites may have several explanations including but not limited to; the duration of time represented by bison enamel (15 to 28 months) and collagen (10-20 years); and, the possibility that the original oxygen and hydrogen isotope values of body water may vary slightly in different biological tissues. More work is needed to fully understand why climatic discrepancies exist between enamel ($\delta^{18}$O) and bone collagen ($\delta$D) isotope values at some archaeological sites and not at others.

Finally, palaeoenvironmental proxies from terrestrial and aquatic environments from southern Saskatchewan were compared to determine whether similar climatic and vegetation conditions are identified during the Holocene. The general congruence of this study indicates that both terrestrial and aquatic proxies have the capability to provide insight into ancient environmental settings. Further, the results of this study also indicate that, when possible, a multidisciplinary approach using both aquatic and terrestrial proxies should be considered based
on the high resolution capabilities of stable isotope analysis on hypsodont teeth (seasonality), and the long record capability of lake-core analyses.
CHAPTER FOUR: SUMMARY AND CONCLUSIONS

For over ten thousand years, people have occupied the Great Plains region of North America. Throughout this time human cultures did not remain static but rather invented and adapted numerous technological traits (e.g. tools, dwelling types, hunting strategies etc.) which allowed them to flourish in often inhospitable environments. For the last century archaeologists have diagnosed and interpreted the lifeways of these early mobile bison hunters by items and features they left behind. Over the last two decades, scientific advancement in the stable isotope analysis of biological tissues (e.g. enamel, bone, and collagen) has shed new light on information we can obtain from the items these early human inhabitants left behind. In this study, I isotopically (carbon, oxygen) analyzed bison tooth enamel carbonate from archaeological sites in southern Saskatchewan for the purpose of inferring climatic and terrestrial habitat conditions which have occurred over the last 9000 year period of the Holocene.

Early studies (DeNiro and Epstein 1978; Longinelli 1984; Luz et al. 1985; Quade et al. 1992) have indicated that a direct correlation exists between the isotope values of an animal’s skeletal tissues and its diet. In the case of large bodied (>100kg) herbivores such as a bison, the general determining factor of carbon ($\delta^{13}C$) isotope values associated with tooth enamel carbonate is the relative abundance of C$_3$ versus C$_4$ grasses consumed during tooth enamel formation. Similarly, the oxygen ($\delta^{18}O$) isotope ratios associated with bison tooth enamel carbonate reflects similar $\delta^{18}O$ values of indigested drinking water. The general dominant factor of meteoric water’s $\delta^{18}O$ value is temperature. Because of this direct correlation, bison tooth enamel carbonate has the capability of inferring potential information regarding the terrestrial habitat (i.e. % of C$_3$ and C$_4$ grasses) and surface temperatures (i.e. climate) of ancient environments occupied during life.

The objectives of this thesis were threefold. First, I sequentially sub-sampled tooth enamel carbonate of nine bison molars ($M_3$) dated to various time periods of the Holocene. A total of eight sub-samples were taken along the horizontal axis of each tooth. I then analyzed the enamel using stable oxygen ($\delta^{18}O$) and ($\delta^{13}C$) isotopes for the purpose of indicating whether $\delta^{18}O$ and $\delta^{13}C$ ratio changes occurred at different section of an individual tooth. Oscillating $\delta^{18}O$ ratios occurring at various locations of an individual tooth would represent temperature/precipitation change (i.e. dietary water source) during tooth enamel formation, thus
indicating climate trends at a seasonal level. Secondly, I compared and contrasted the mean oxygen (δ\(^{18}\)O) and carbon (δ\(^{13}\)C) isotope ratios of tooth enamel with the mean hydrogen (δD) and carbon (δ\(^{13}\)C) isotope ratios of bone collagen (Leyden 2004) from fossil bison remains from the same eight archaeological sites. The reason why I compared the isotope ratios of bison enamel and collagen was to identify whether these two types of bison skeletal tissues would indicate similar climate and terrestrial habitats at the same time periods of the Holocene.

Thirdly, I took the mean δ\(^{18}\)O and δ\(^{13}\)C values from the eight teeth representing various time periods of the Holocene and compared the enamel results against various aquatic palaeoenvironmental proxy studies previously done in the northern Great Plains.

The δ\(^{18}\)O and δ\(^{13}\)C isotope results from the enamel sub-samples of all eight of the teeth analyzed in this study produced a similar sinusoidal curve, which I interpret is a product of surface water temperature (seasonality) change, and change in the amount of C\(_3\) and C\(_4\) grasses occurring during tooth formation. It is assumed that the enamel sub-samples with the lowest \(^{18}\)O and highest δ\(^{13}\)C values represent the winter months, whereas the highest \(^{18}\)O and δ\(^{13}\)C lowest values are associated with the moist growing seasons months.

The comparison of the stable isotopes of tooth enamel (δ\(^{18}\)O and δ\(^{13}\)C) and bone collagen (δD and δ\(^{13}\)C) of bison from eight archaeological sites in Saskatchewan produced mixed results. The carbon isotopes compositions from both types of skeletal tissue produced comparable results regarding the abundance of C\(_3\)/C\(_4\) grasses consumed by bison populations at each particular time period of the Holocene. Evidence from the carbon isotope data in this study present two possibilities; one, that carbon isotope data from both enamel and collagen provide similar information from which to derive models for reconstructing diet in archaeological bison populations; two, carbon isotope ratios from both biological tissues have the potential of detecting habitation shifts (i.e. C\(_3\) versus C\(_4\) grass consumption) produced by temperature and precipitation levels at particular time periods on the northern Great Plains.

Palaeoclimatic proxy results from bison tooth enamel (oxygen) and bone collagen (hydrogen) isotope analysis in this study produced variable results. Interestingly, comparable climatic trends were indicated by both enamel and collagen at several time periods but considerably different climatic trends were detected at others. This discrepancy between the collagen and enamel results has forced the author to consider several possible explanations.
including; the time difference represented by the bison enamel M\textsubscript{3} (15 to 18 months) and bone collagen (10-20 years); and, lack of experimental studies conducted on biological tissues of large herbivores and hydrogen isotope compositions may hinder our knowledge on the climatic data determined from bone collagen.

Results from the mean δ\textsuperscript{18}O and δ\textsuperscript{13}C values of the eight bison teeth analyzed in this study appears generally comparable to climate condition indicated by various other aquatic based proxy studies conducted on the northern Great Plains. Two notable late-Holocene climate trends were detected by the mean δ\textsuperscript{18}O and δ\textsuperscript{13}C results from the Fitzgerald (~1450BP) and Tschetter (~1050BP) which have been detected by other palaeoenvironmental proxies but at slightly different time periods. The Fitzgerald mean δ\textsuperscript{18}O and δ\textsuperscript{13}C values indicate a dramatic increase in temperature when compared to the rest of the time periods represented in this study. Interestingly, this warm climatic trend has been detected by various other proxies but at a later time (1100 to 800 years BP) period (Teller and Last 1990; Vance et al., 1997). Similarly, the mean δ\textsuperscript{18}O and δ\textsuperscript{13}C results from the Tschetter tooth indicate a dramatic cool and humid trend in the late-Holocene. Various other palaeoenvironmental proxy studies (Vance et al., 1993 and 1997) conducted on the northern Great Plains have detected this cool and humid trend but also at a later time period (500 to 100 years BP). I suggest that this time delay seen between the δ\textsuperscript{18}O and δ\textsuperscript{13}C values of tooth enamel and the aquatic palaeoenvironmental proxies maybe represent a time delay which may occur between terrestrial and aquatic research methods.

I suggest that a further experimental study needs to be conducted on the relationship of hydrogen and oxygen isotope compositions of tooth enamel and bone collagen of modern bison populations to further understand the discrepancies seen in this study. The capability of detecting temperature change at a seasonal level in bison tooth enamel sub-samples and the long period (10-20 years) of averaged climate and dietary consumption associated with bison collagen would present a detailed picture of palaeoenvironmental conditions at ancient time periods. Together, isotope analysis of both biological tissues of bison has the potential to generate invaluable information on environmental settings at specific time periods.
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Appendix A: BRIEF OVERVIEW OF ARCHAEOLOGY SITES AFFILIATED IN THIS RESEARCH PAPER

A.1 Introduction

The bison teeth isotopically analyzed in this study are from eight archaeological sites previously and independently excavated from the province of Saskatchewan. The archaeological sites used in this study were based on a sample collection previously selected by Jeremy Leyden (2004). Leyden (2004), isotopically (hydrogen, carbon and nitrogen) analyzed bone collagen from fossil bison remains associated with these eight archaeology sites for the purpose of creating a palaeoenvironmental record of southern Saskatchewan as well as studying possible changes in bison foraging behaviour during the last 9000 years of the Holocene.

Several of the archaeological sites used in this study are multi-component and/or multi-occupational. In this appendix, archaeological site discussions will be limited to the cultural levels which bison teeth were collected from and used in this study. Table C.1 includes radiocarbon dates as well as the cultural affiliation associated with each archaeological site and/or occupational level used in this study. Table A.1 has been added to incorporate the cultural history of the northern Great Plains based on diagnostic projectile points. Figure A.1 is a map which illustrates the general distribution of the archeological sites utilized in this thesis. The following is a brief description of these archaeology sites. The order that the sites are presented are from oldest to youngest in age according to radiocarbon dates in Linnamae (1988), Zurburg (1991) and Leyden (2004).
Table A.1 Prehistoric Cultural Chronology on the Northern Great Plains Based on Diagnostic Stone Technologies

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<td>Cody Complex</td>
<td>8,600 – 9,500</td>
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<td>7,500 – 8,500</td>
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<tr>
<td>Prairie Side-notched</td>
<td>1,200 - 800</td>
</tr>
<tr>
<td>Plains Side-notched</td>
<td>550 - 250</td>
</tr>
</tbody>
</table>

All dates used in Table A.1 have been adapted from Walker (1992)
Figure A.1 Map of archaeological sites used in this thesis
A.2 The Heron Eden site (EeOi -11)

The Heron Eden site is located on a small knoll in a cultivated field approximately 13km south of Prelate, Saskatchewan (Corbeil, 1995). In a regional context, the Heron Eden site is situated near the Great Sand Hills and has a modern vegetation cover of semi-arid grasslands. Located in southwestern Saskatchewan, this archaeological site is identified as the most northerly located Cody Complex site on the Great Plains (Corbeil, 1995).

Based on the high number (n=36) of individual bison (probably *Bison bison antiquus*) represented along with the severe degree of bone fragmentation, the Heron Eden site has been interpreted as a single component bison kill and butchering site (Corbeil 1995). Contents found in the excavated portion of the Heron Eden site included lithic artifacts, including 14 projectile points, eight of which were culturally identifiable, a bifacial chopper, a burin, several uniface tools as well as several re-touched flakes (Corbeil 1995). The lithic materials used to produce the artifacts associated with the Heron Eden site are a mixture of local and imported stone (Corbeil 1995). Faunal remains (n=220,164) excavated from the site are predominantly those of bison with only 11 bone specimens identified as non-bison (Corbeil 1995).

The absolute age of the Heron Eden site was determined through radiocarbon dating of five unburned bison bones (Corbeil 1995). Of the 5 submitted bone samples, three dates were approximalty 9000 years BP which is within the accepted radiocarbon dates associated with the Cody complex. Herd dynamic research conducted through dental eruption patterning of juvenile specimens, tooth wear patterning, and sexual identification analyzed through long bone measurements suggest that the Heron Eden site was occupied in the winter, more specifically in the months of December/January (Corbeil 1995).

Of the eight identified projectile points found at the Heron Eden site only three were found *in situ* and have been identified as Scottsbluff type points (Corbeil 1995). The remaining five were surface collected with four of them diagnosed as Scottsbluff with the other representative of an Eden type point. All identifiable projectile points associated with this site are affiliated to the Cody Complex, a Palaeoindian big game hunter/gather cultural group which occupied the Great Plains from ~9,500 to 8,750 years BP (Frison 1998).

Due to the presence of two groupings (males and female/immature) in the Heron Eden
bone bed, Corbeil (1995) suggests that the site may represent two kill/butchery events, one which included a bull herd and the other included a mixed cow and calf herd both of which occurred in the winter. Site topography along with no physical evidence of posthole features provides little evidence as to what hunting method may have been used at the Heron Eden site. The accumulation of stone tools as well as the skeletal dispersal and skeletal elements present in the undisturbed portion of the bone bed suggests that the Heron Eden site was a multi-functional site.

A.3 The Gowen sites (FaNq -25) and (FaNq – 32)

Located on the southwest edge of the city of Saskatoon landfill, the discovery of Gowen 1 occurred in 1977 followed by the discovery of Gowen 2 in 1980 (Walker 1992). The contents of both Gowen sites were exposed by subsurface excavation of work crews of the landfill, including the discoverer Charlie Gowen (Walker 1992). Excavations of the Gowen sites were conducted under the supervision of the Department of Anthropology and Archaeology of the University of Saskatchewan.

The Gowen sites were situated approximately 70 meters apart from each other and both contained a single occupational level (Walker 1992). Extensive number (98) of projectile points were recovered from the Gowen sites including numerous diagnostic (e.g. Mount Albion Corner-notched, Bitteroot Side-notched and Blackwater Side-notched) points affiliated to the Mummy Cave complex (Walker 1988). The site included a previously unrecorded side-notched projectile point, which is now referred to as the Gowen Side-notched point (Walker 1992). Although Walker (1992) described the new point as unique, he concluded that the points had some characteristics of projectile points associated with the Mummy Cave complex.

Faunal remains from the Gowen sites were largely highly fragmented bison bone (Walker 1992). Due to the highly fragmented collection of bison bone, the presence of anvil stones and several pit and hearth features, Walker (1992) suggested food availability for the occupants of the Gowen sites may have been poor. A total of nine radiocarbon dates were taken from the Gowen sites, with the majority of the dates (eight) occurring around 6000 radiocarbon years (Walker 1992). The similarities (e.g. radiocarbon dates, artifacts, faunal assemblage) observed between the two Gowen sites suggests that the sites were contemporaneous with each other and generally represent a short-term bison hunting and processing camp (Walker 1992).
A.4 The Norby site (FaNq-25)

The Norby site is located within the city of Saskatoon, on the 900 block of Avenue M south (Zurburg 1991). The Norby site was initially discovered during subsurface excavations for a residential basement in 1988 (Zurburg 1991). Les Norby (owner) contacted the Anthropology and Archaeology Department at the University of Saskatchewan to determine the importance of the archaeological find. Test excavations determined that the Norby site was of significant importance due to its relatively early age. A full scale excavation was conducted and completed during the 1989 field season (Zurburg 1991).

Excavations recovered four projectile points, three of which were diagnosed as Gowen side-notched points with the fourth determined to be a possible Late Palaeoindian point (Zurburg 1991). A total of three radiocarbon dates were established from bison bone associated with the Norby site which determined the site to be around 5,800 years BP (Zurburg 1991). Given the radiocarbon dates and the diagnostic projectile points, Zurburg (1991) determined the Norby site to be that of the Mummy Cave complex. It is worth mentioning at this point that Leyden (2004) re-dated the Norby site using the AMS dating technique and recorded a considerably (>1200 yrs) earlier date then those attained by Zurburg (1991). See Appendix C of this thesis for discussion regarding this issue.

The majority of the faunal materials associated with the Norby site were highly fragmented bison bone (Zurburg 1991). Established through the analysis of bison central fused and fourth tarsals, the MNI at the Norby site is assumed to include the skeletal remains of 26 individuals (Zurburg 1991). Through the use of bivariate plots of several measured bison elements (long bone, carpals and tarsals), Zurburg (1991) determined that the bison population at the Norby site was predominantly male. Given the small number of formed tools (20) and the high number of bison specimens, the Norby site is interpreted to be a bison kill/butchering site (Zurburg 1991). An important observation is made by Zurburg (1991) which suggests that the bison associated with the Norby site may have been a transitional species referred to as *Bison bison occidentalis*.

A.5 The Amisk site (FaNq-17)

The Amisk site is located in the Wanuskewin Heritage Park, which is located
approximately 2.5 kilometers north of the city of Saskatoon. First identified through an archaeological resource assessment of the park in 1985, the Amisk site was later excavated for academic purposes through the field seasons of 1984 and 1985 (Amundson 1986). The Amisk site is a multi-component campsite which consists of seven occupational levels, only three of which produced diagnostics of known cultural complexes (Amundson 1986). Level one contained projectile points recognized as Plain Side-notched, Prairie Side-notched and Avonlea styled points (Amundson 1986). Diagnostic projectile points associated with levels four and five of the Amisk site have been described as Oxbow points (Amundson 1986). Radiocarbon dates obtained from bison bone of levels four and five from the Amisk site are 4,015±195 and 4,120±190 radiocarbon years, respectively (Amundson 1986).

Faunal remains associated with the fourth level of the Amisk site included bison bone featuring cut marks and fragmentation, a possible sign of butchering (Amundson 1986). In addition, a larger amount of lithic tools, cores and debitage was associated with the fourth level suggesting tool manufacturing occurred at the Amisk site (Amundson 1986). The combination of worked faunal remains, tool manufacturing, and a large fire hearth signifies that level four at the Amisk site was mostly likely an Oxbow campsite (Amundson 1986).

**A.6 The Harder Site (FbNs – 1)**

Four years (1969, 1970, 1971, 1972) of site excavations in conjunction with the collection of material remains and the presence of numerous features suggests that the Harder site represents a prehistoric bison hunters’ campsite affiliated to the Oxbow complex (Dyck 1977). At the time of excavation, the Harder site was located in a shallow depression in the Dunfermline Sand Hills, approximately 33 km north west of Saskatoon, with vegetation cover dominated by aspen stands (Dyck 1977). The Harder site was located approximately 18 km south of the North Saskatchewan River and 23 km north-west of the South Saskatchewan River (Dyck 1977). At a regional scale, the sand hills which house the Harder site are surrounded by a northern plains type environment (Dyck 1977). Almost completely under cultivation, a historical record (Dominion Land Survey) of this region indicates a vegetation regime characterized by prairie grasses with small aspen and willow bluffs (Dyck 1977). Dyck (1977) indicates that the Harder site may have been utilized by bison and nomadic hunters for shelter during treacherous winter conditions on the adjacent barren plains.
The stratigraphic layer which incorporated the Oxbow occupational material was described as a grey-black sandy soil ranging in thickness from 12 to 28 cm with the start of the zone approximately 30 cm below the present surface (Dyck 1977). Cultural material situated within this stratigraphic layer included chipped stone tools including but not limited to several large bifaces, uniface knives, hammers, anvils, retouched flakes as well as twenty four complete side-notched projectile points recognized as Oxbow type (Dyck 1977). The majority of the faunal remains recovered from the Harder site was heavily pulverized bison bones (Dyck 1977). Quantitative analysis using bison left tibia was conducted by Dyck (1977) who indicated a MNI of 17 individuals. Notable features at the Harder site included fire hearths, smudge pits, refuse area and the possibility of recognizable dwelling floors (Dyck 1977).

Bison bone was submitted on two separate occasions for the purpose of absolute dating the Harder site. Initial dates for the site were obtained by Dyck (1977) who established two un-calibrated radiocarbon dates of 3,360±120 and 3,425±105 radiocarbon years. Dyck (1977) noted that these radiocarbon dates appear to be around 1000 years younger than the majority of Oxbow site in the area. Several years later, Morlan (1994) re-examined the Harder site and submitted three additional bison bone specimens for dating. Dates from the Harder site were 3,420±140, 4,410±150 and 4,190±90 radiocarbon years (Morlan 1994). The two older dates fit within the general acceptance of the Oxbow culture. Morlan (1994) suggests that the younger dates associated with his sampled specimen and the dates earlier obtained by Dyck may indicate the possibilities of post-mortem contamination (diagentic effects) of bison bone.

A.7 The Thundercloud site (FbNp-25)

Located on the northern edge of the Wanuskewin Heritage Park, the Thundercloud site was initially discovered during an archaeological assessment of the park in 1982 (Webster 1999). Years later, excavations were conducted at the Thundercloud site under the supervision of the Department of Anthropology and Archaeology at the University of Saskatchewan for academic purposes. Full scale excavations of the site revealed seven soil horizons as well as ten different cultural occupation levels (Webster 1999).

Numerous prehistoric cultural diagnostics were unearthed in level one of the Thundercloud site including Prairie Side-notch, Plains Triangular and Plains Side-notched
projectile points (Webster 1999). Stratigraphic mixing was evident between levels two and three causing a wide array of diagnostics including Prairie Side-notched, Plains Triangular, Plains Side-notched, Avonlea and Besant projectile points (Webster 1999). Level four was described by Webster (1999) as a sterile layer which produced no cultural material. Level five of the Thundercloud site contained cultural material including numerous projectile points (e.g. Duncan, Hanna, McKean lancelot) designated to the McKean Cultural Complex (Webster 1999). An uncalibrated radiocarbon date of 4040±90 BP was obtained from level five (Webster 1999). Level six of the Thundercloud site included Oxbow styled projectile points whereas level seven contained no diagnostic materials (Webster 1999).

Faunal remains recovered from the Thundercloud site was predominantly bison. The MNI counts indicated that at least three bison individuals were present in all soil horizons with the exception of level seven (Webster 1999). Features associated with the site included hearths, soil stains and areas which featured high quantities of processed bison bone (Webster 1999). Webster (1999) noted that the combination of cultural material, features and processed faunal remains associated with level five provides evidence that Thundercloud was most likely used as a processing area/camp site.

A.8 The Fitzgerald site (ElNp-8)

Located approximately 15 kilometers southeast of Saskatoon, the Fitzgerald site is considered to be a Besant bison pound and processing area (Hjermstad 1996). Discovered by landowners (surname Fitzgerald) in 1991, excavations at the Fitzgerald site occurred in the summers of 1992 and 1993 under the supervision of the Department of Anthropology and Archaeology at the University of Saskatchewan (Hjermstad 1996). The two summers of excavation produced an extensive amount of bison bone including a MNI count of 49 (Hjermstad 1996). Lithic material included numerous projectile points (143 complete/partial) as well as 22 other formed tools (e.g. end scrapers, bifaces, unifaces) (Hjermstad 1996). In addition, three ceramic shards were discovered at the Fitzgerald site, unfortunately Hjermstad (1996), noted that the ceramics were not indicative of a specific cultural ware.

A unique array of features were associated with the Fitzgerald site including post molds, soil stains and bone uprights, all considered to represent a bison pound (Hjermstad 1996). Other
recognizable features included a basin like pit as well as an area of highly compact bison bone. Hjermstad (1996) suggests that the pit and dense bone area most likely represents the processing location. A total of four bison bone specimens were submitted for radiocarbon dating (Hjermstad 1996). The mean of the four dates was around 1,300 year BP, an acceptable date for the Besant culture in the geographic area (Hjermstad 1996)

A.9 The Tschetter Site (FbNr -1)

The Tschetter site is described as being a single component site occupied by Late Prehistoric bison hunters and developed for the purpose of impounding, killing and butchering bison (*Bison bison*) (Prentice 1983). Cultural material contained within the Tschetter site include numerous lithic tool types such as projectile points, endscrapers, bifaces, unifaces, drills, perforators, spokeshaves and retouched flakes all of which were predominantly produced from local material (Prentice 1983). Several pottery fragments representing 4 different vessels were also associated with the Tschetter site (Linnamae 1988). Faunal remains associated with the Tschetter site are predominantly (99 %) bison with an estimated individual count of 96 (Prentice 1983). Features within the Tschetter site include a bison bone bed, post holes, two pits, and the presence of charcoal (Prentice 1983).

The Tschetter site is located in the northeastern corner of the Dunfermline Sand Hills approximately 16 km west and 10 km north of Saskatoon in the province of Saskatchewan and is situated in the Saskatchewan River Plains and within the Aspen Parkland ecological region (Prentice 1983). The sandy soil and topography of the Dunfermline Sand Hills support grasslands and small stands of aspen (*Populus tremuloides*) (Prentice 1983). As an ecotone, the Aspen Parkland region is a transition zone between the grassland prairies to the south and the boreal forest to the north.

Three absolute dates have been acquired from bone collagen of bison specimens from the Tschetter site bone bed and are as follows; 1,000 ± 75, 1,020 ± 100 and 915 ± 45 radiocarbon years BP (Linnamae 1988). Tooth wear and eruption analysis conducted on bison mandibles, as well as the distant location from a permanent water source would suggest that the Tschetter site was occupied during the winter months (Prentice 1983). Projectile points excavated from the Tschetter site are identified as Prairie Side-Notched (Late Side-Notched tradition) form and are
generally associated with the “Old Women’s”, a cultural complex known to manufacture vessel pottery (Prentice 1983; Linnamae 1988). The topography, presence of post holes and the bone bed suggest that the Tschetter site was a winter bison pound (Prentice 1983; Linnamae 1988). Tool types, charcoal, pit features, highly pulverized bone and the lack of long bones present in the site have been interpreted that the major purpose of the Tschetter site may have been bone marrow and grease extraction (Linnamae 1988).
Appendix B: MATERIALS AND METHODOLOGY

B.1 Materials

Bison continuously form teeth until ca. 2 years of age (Gadbury et al. 2000). Bison have three mandibular molars that are generally referred to as M₁, M₂ and M₃. The crown of M₁ in bison forms partially en utero and is completely developed several months after birth (Fricke and O’Neil 1996; Gadbury et al. 2000). The crown of M₂ begins forming shortly after birth and completes development at approximately ca. 13 months of age (Fricke and O’Neil 1996; Gadbury et al. 2000). The crown of M₃ generally begins forming nine months after birth and is completely formed when an animal is ca. 2 years of age (Fricke and O’Neil 1996; Gadbury et al. 2000). Because of the different developmental periods associated with each bison mandibular tooth, it is generally understood (Fricke and O’Neil 1996; Gadbury et al. 2000; Balasse 2002) that isotopic compositions obtained from a particular tooth will not reflect the same dietary intact as the other. In order to make isotope analysis of tooth enamel carbonate more meaningful, we first must understand the dietary inputs of particular teeth.

As previously mentioned, a bison M₁ is formed partially en utero. Because mammalian fetuses are nourished in the womb by the blood of the mother, isotope data obtained from this tooth is potentially influenced by the mothers diet (Balasse 2002). Suggestions have been made (Kohn et al. 1996; Bryant et al. 1996; Gadbury et al. 2000) that carbon and oxygen values ingested by the fetus may not represent the mother’s bulk diet. In other words, developing fetuses may have a fractionation factor of biosynthesis which is currently unknown, or making it difficult to interpret (Gadbury et al. 2000). Like M₁, isotope values obtained from the enamel carbonate of bison’s M₂ may also be influenced by maternal biosynthesis. The crown of a bison’s M₂ is predominantly formed during the period of time the bison calf is nursing/suckling. Experimental studies (e.g. Balasse 2002) conducted on enamel carbonate of the M₂ of steers (Bos taurus) has indicated that isotope values of this tooth are influenced by the consumption of the mother’s milk and may mix original isotope signals as much as ~5‰. On the contrary, the crown of a bison’s M₃ forms almost entirely post-weaning. Physiological and metabolic rates (see Fricke et al. 1998; Bryant and Froelich 1995 for detailed discussion) are constant throughout amelogenesis (tooth enamel formation) of M₃, therefore isotopic compositions obtained from the enamel should yield the most direct evidence of an adult diet.
Enamel carbonate used in this study was collected from fully developed and well preserved fossil bison M₃ teeth. Teeth selected in this study had a crown height between 50 and 53 mm, thus representing teeth of minimal wear. All teeth analyzed in this study were previously and independently excavated (*in situ*) from prehistoric archaeological site in Saskatchewan. One tooth was analyzed from each of the nine archaeological sites previously described in Appendix A of this thesis. In addition, each tooth is associated with a radiocarbon date (see Appendix C of this study) obtained from bison skeletal (collagen) remains of each archaeological site. In the presence of multi-component/cultural sites, radiocarbon dates represent the level in which teeth were extracted from.

B.2 Methodology

B.2.1 Introduction

The methodology used in this study was based on numerous studies (e.g. Fricke and O’Neil 1996; Gadbury et al. 2000; Balasse 2002; Zazzo et al. 2002; Hoppe 2006; Bernard et al. 2009) which successfully analyzed tooth enamel carbonate of hypsodont teeth for isotopic analysis. This methodology has been tested in several controlled feeding studies (Fricke and O’Neal 1996; Balasse 2002; Zazzo et al. 2006) on bovine tooth carbonate and results have proven to be consistently accurate in correlating isotope values with consumed diet. The following is a brief presentation on the sampling techniques, pretreatment method, and mass spectrometry used in this thesis.

B.2.2 Intra-tooth sub sampling

Early research (e.g. Sharp and Cerling 1998; Cerling and Sharp 1996; Fricke and O’Neil, 1996; Fricke et al. 1998) of isotope analysis on hypsodont tooth enamel recognized that teeth form from the apex (crown) to the cervix (root). Because of this growth sequence, the oldest or first lain enamel of a bison M₃ will form the crown and will progressively develop down the axis of the tooth. For this reason, isotope values associated with each section of a bison’s tooth will correlate with dietary changes and should thus reflect seasonal dietary (food and water) availability during the ca. 2 year growth of the tooth.

Prior to enamel collection, each tooth was vigorously washed with distilled water (3X)
and set aside to dry. Each tooth was then manually cleaned with stainless steel dental tools followed by a light buffering with a Dremel™ tool with a sanding drum attachment for the purpose of removing any calculus or foreign material adhering to the enamel surface. Once pristine enamel was exposed, each tooth was then rewashed (3X) with distilled water and set aside to dry overnight. Using a Dremel™ with a diamond drill bit, enamel powder samples were collected from the metaconid cusp of the tooth in approximately 3mm wide bands perpendicular to the growth axis and through the entire thickness of the enamel stopping at the CEJ (cementum-enamel junction). Enamel carbonate was collected from eight separate locations at approximately 5-6 mm intervals (Figure B.1). Powdered enamel was collected from weigh paper and placed into test tubes.

**Figure B.1** Schematic of Enamel Carbonate Sampling Protocol used in This Study

B.2.3 Enamel Carbonate Isotope (δ¹⁸O and δ¹³C) Analysis

* B.2.4.A Possible pretreatment effects
The pretreatment of enamel carbonate procedure used in this study is comparable to Zazzo et al. (2005). I have chosen this method over the conventional (Koch et al. 1997) method for several reasons. First, Koch et al. (1997) conducted an exhaustive experimental study on the isotope value differences obtained from untreated and treated tooth enamel carbonate. In Koch et al. (1997), half of tooth enamel samples were treated for the purpose of organic material removal with NaOC1 solution followed by a weak acetic acid (0.1N) buffer bath for the diagenetic carbonate removal prior to mass spectrometry analysis. The other half of the enamel carbonate was placed directly into the mass spectrometry for isotopic analysis. Results from Koch et al. (1997) did not find significant isotopic data differences in treated and untreated enamel carbonate.

In a second experimental study, Zazzo et al. (2006) measured the effects of untreated and treated (same treatment method as Koch et al. 1997) dentine (20% organic material) and found significant differences in carbon isotope values between the two. Zazzo et al. (2006) suggested that the untreated dentine samples are more reliable than the treated samples and further, indicates that bleaching make fractionate dietary isotope values as much as 4-5‰ per sample. Also, because AMS dates affiliated with each of the nine archaeological site were deemed acceptable (no signs of diagenetic alteration) and the observational condition of the teeth enamel was characterized as pristine, the author felt chemical pretreatment presented a greater chance of creating isotope offsets than untreated samples. Finally, it has been recognized (Koch et al. 1997) that if extreme consistency in pretreatment solutions as well as the constant duration of each sample in the pretreatment solution bath is not accurate, original isotope data may be modified. In addition, repeated rinsing is required to remove all bleach residues, each washed powdered carbonate sample results in the loss of finer suspended materials which may also modify the original isotopic value of a carbonate sample.

**B.2.4.B Pretreatment procedure and mass spectrometry used in this study**

Powdered enamel carbonate is roasted in a vacuum oven at 200°C for 1 hour to remove water and volatile organic contaminants that may alter stable isotopes values of carbonates. Using a Finnigan Kiel-IV carbonate preparation device, approximately 50 micrograms of carbonate was reacted at 70°C with 3 drops of anhydrous phosphoric acid for 420 seconds. The
CO₂ evolved is then cryogenically purified before being directly passed on to the dual inlet of a Finnigan MAT 253 isotope ratio mass spectrometer housed in the Saskatchewan Isotope Laboratory located at the University of Saskatchewan. Isotope ratios are corrected for acid fractionation and $^{17}$O contribution using Craig correction (Coplen 1995), and reported using standard delta (δ) and in per mill (‰) notations relative to VPDB scale.

Data is directly calibrated against the international standard NBS-19 that is by definition $\delta^{13}$C = 1.95‰ V-PDB and $\delta^{18}$O = -2.2‰ V-PDB. $\delta^{18}$O samples are also reported in V-SMOW using the Coplen (1995) equation where; $\delta^{18}$O V-SMOW=1.03092 x $\delta^{18}$O V-PDB+30.92.

Precision/accuracy of data are monitored through routine analysis of NBS-19 and in-house check standards which have been stringently calibrated against NBS-19. Precision/accuracy of $\delta^{13}$C and $\delta^{18}$O are 0.0 and 0.11‰, respectively (n=25). The results of the isotopic analyses are expressed:

$$\delta^{13}C = [(^{13}C/^{12}C_{sample}/^{13}C/^{12}C_{standard}-1) \times 1000 \, \text{‰} \, \text{(V-PDB)}}$$

$$\delta^{18}O = [(^{18}O/^{17}O_{sample}/^{18}O/^{17}O_{standard}-1) \times 1000 \, \text{‰} \, \text{(V-SMOW)}}$$
Appendix C. RADIOCARBON DATES

C.1 Introduction

All radiocarbon dates referred to in chapters two and three of this thesis have been attained from the previous and independent work of Leyden (2004), Zurburg (1991) and Linnamae (1988). Leyden (2004) had bison bone collagen from all archaeological sites used in this study sent to the Rafter Radiocarbon Laboratory in New Zealand for accelerator mass spectrometry (AMS) radiocarbon dating. The results of the AMS dating associated with the eight archaeological sites are reported in Table C.1. Leyden (2004) reported the AMS dates obtained from the Tschetter site were erroneous, yielding values too recent in age, and therefore were discarded from this study. Therefore, the absolute date associated with the Tschetter site is an aggregated mean radiometric radiocarbon date previously determined by Linnamae (1988); and is reported in Table C.1.

The AMS date (Leyden 2004) for the Norby site is considerably (>1200 yrs) earlier than the original three radiocarbon dates obtained by Zurburg (1991). Further, three identifiable Mummy Cave projectile points were associated with the submitted material (unburnt bone) for dating of the Norby site. The original dates (Zurburg 1991) correspond well with previously established Mummy Cave projectile point chronology; therefore, an aggregate mean radiometric date previously determined by Zurburg (1991) was chosen to represent the Norby site.
Table C.1 Original and Secondary Radiocarbon Dates Associated With Archaeological Sites Used In This Thesis

<table>
<thead>
<tr>
<th>Site</th>
<th>Heron Eden</th>
<th>Gowen</th>
<th>Norby</th>
<th>Amisk</th>
<th>Harder</th>
<th>Thundercloud</th>
<th>Fitzgerald</th>
<th>Tschetter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Borden #</td>
<td>EeOi-11</td>
<td>FaNq-25/32</td>
<td>FaNq-56</td>
<td>FbNp-17</td>
<td>FbNs-1</td>
<td>FbNp-25</td>
<td>ElNp-8</td>
<td>FbNr-1</td>
</tr>
<tr>
<td>Cultural Affiliation</td>
<td>Cody Complex (Eden)</td>
<td>Mummy Cave (Gowen)</td>
<td>Mummy Cave (Gowen)</td>
<td>Oxbow</td>
<td>Oxbow</td>
<td>McKean (Level 5)</td>
<td>Besant</td>
<td>Oldwoman’s Complex (Prairie Side-notched)</td>
</tr>
<tr>
<td>Radiocarbon Age Yrs. BP (Normalized)</td>
<td>9168 ± 50¹</td>
<td>5863 ± 53 5566 ± 143*</td>
<td>5808 ± 33³</td>
<td>4358 ± 45¹</td>
<td>4221 ± 45¹</td>
<td>3383 ± 55¹</td>
<td>1563 ± 45¹</td>
<td>1035 ± 40²</td>
</tr>
<tr>
<td>Calibrated Range Yrs. BP (1Sigma)</td>
<td>10352 ± 134*</td>
<td>6653 ± 143*</td>
<td>7846 ± 103*</td>
<td>4941 ± 103*</td>
<td>4823 ± 33*</td>
<td>3804 ± 13*</td>
<td>1442 ± 101*</td>
<td></td>
</tr>
</tbody>
</table>

²Radiocarbon Age Yrs. BP (Normalized) from Leyden (2004)
³Averaged radiometric radiocarbon dates determined by Linnamae (1988)
⁴Averaged radiometric radiocarbon dates determined by Zurburg (1991)
*Calibrated Range Yrs. BP (1 Sigma) from Leyden (2004)