PALEOECOLOGY OF SOUTHERN SASKATCHEWAN BISON: CHANGES IN DIET AND ENVIRONMENT AS INFERRED THROUGH STABLE ISOTOPE ANALYSIS OF BONE COLLAGEN

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By
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ABSTRACT

Archaeological research has provided evidence of change in the settlement and subsistence practices of human groups inhabiting the Great Plains throughout the Holocene. A substantial part of this reorganization appears to be tied to concurrent changes affecting local bison populations, a species upon which these groups were uniquely dependant. Although bison are thought to have been strongly affected by the severe climates of the Mid-Holocene, there is an absence of appropriate models from which to interpret data in the archaeological and paleontological records. Nevertheless, new techniques are allowing for the determination of ecological information directly from prehistoric remains. This study uses stable isotope ratios ($\delta^{13}C$, $\delta^{15}N$, δD) in bone collagen to examine the dietary ecology of bison in Southern Saskatchewan during eight distinct time periods spanning the last 9,000 years. Stable isotopes of hydrogen and nitrogen in the tissues of animals relate to aspects of local climate, while stable-carbon isotope values reflect dietary choices. When employed in a comparative fashion, these sources may be used to construct simple models of foraging behaviour.

The environmental data developed from this investigation appear to correlate generally with patterns predicted by conventional models of Holocene climate. Nevertheless, at least one period of unexpectedly moderate temperature was identified from a context dating to the arid Mid-Holocene. The ecological impact of such an episode may have been significant. In addition, the results of this study suggest that bison diet has a complex relationship with local climate. Changes in plant distribution resulting from variations of temperature and precipitation appear to have less of an impact upon bison consumption patterns than do climatically induced changes in the nutritional quality of vegetation. Nevertheless, during specific time periods characterized by similar climatic regimes, their relative consumption of certain forage species (C_3 and C_4 plants) does not appear to have been consistent. Such a discrepancy may reflect adaptive differences between bison from distinct time periods, or alternatively, the effects of a climatic difference undetectable by isotopic means. In either case, it would appear that bison of the past may have been subject to significant

nutritional stresses that could have caused them to behave in fundamentally different ways from modern populations.

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Chapter 1. BISON BONE IN ARCHAEOLGICAL AND PALEOECOLOGICAL STUDIES

1.1 Introduction

Prior to European contact, the most abundant large mammal in North America was the American Bison (*Bison bison bison)*. With a continental population estimated to have be as high as 70 million, large herds of hundreds of thousands of individuals were reported in parts of the Great Plains, although by 1889 numbers were reduced to less than 1,000 (Jones *et al.* 1983). Bison, by virtue of their size, numbers and behaviour were integral agents within the region's ecosystem. Their grazing and movements both altered and promoted the landscapes over which they ranged, impacting themselves and other species (Larson 1940; Vinton *et al.* 1993; Griebel *et al.*1998; Truett *et al.* 2001).

From the end of the Pleistocene, through much of the Holocene, bison were the predominant "game" animal and primary resource for the indigenous peoples of the Great Plains. The often extreme nature of their environment led these peoples to develop a practical and conservative culture in which resources were utilized to an astonishing degree. Almost all parts of the bison including its hide, bones, horns, hair, tendons, bladder, scrotum and even its dried dung were used for consumption, rationing, clothing, bedding, shelter, fuel, tools and objects of ritualistic and ceremonial importance (Roe 1951; Bryan 1991). This economic dependence fostered the social, ideological and cultural importance of bison to prehistoric peoples. Bison social and foraging behaviour dictated the movements of human groups and may even have been an important factor in the persistence of a nomadic hunting and gathering subsistence seen in most Great Plains peoples throughout the Holocene (Verbicky-Todd 1984; Frison 1991).

Understanding the pre-contact ecology of bison is essential for archaeologists and paleoecologists engaged in Great Plains research. Difficult as the task of understanding the ecology of extant taxa might be, bison studies are further complicated

by the near extinction of the species which has resulted in vastly reduced numbers and the existence of only a few non-captive herds (Jones *et al.* 1983). Those herds that are largely unmanaged usually occur within national parks and other protected regions, and are restricted in terms of range and movement. By far, however, the vast majority of extant bison occur in private managed herds, many of which service the growing commercial bison industry (McDonald 2001). These individuals, while far less managed than commercial cattle, often survive on supplemented diets in comparatively restricted habitats. As a result, the modern researcher of natural bison ecology faces a task complicated by the near extermination and subsequent restoration of the species along with resulting alterations in genetic diversity and population distribution (Garretson 1938; Truett *et al.* 2001). In addition, human generated and natural environmental changes have continued to alter Holocene climates and ecosystems within the Great Plains, as well as both continentally and worldwide. Even if modern bison had been sustained in terms of frequency, distribution, biology and behaviour, it is quite possible that their current habitats are no longer analogous to those of the past.

Despite these limitations, modern studies of natural bison ecology have followed several conventional approaches. Observations of living individuals and herds have provided analogy through which the biology and behaviour of past animals may be inferred and compared (Bamforth 1988; Frison 1991; Larson *et al.* 2001). This information comes primarily through those who raise bison and manage herds for commercial, ecological and scientific purposes. Another approach involves the difficult and often conflicting analysis of historical records from the time of European contact through the subsequent colonization of North America. Historical records have been the source for much of our demographic knowledge of precontact herds including their behaviour and relationships with indigenous peoples (Cannon 2001). Despite their utility, these records, more often than not, contain inaccuracies which may be obvious or insidious, intentional or accidental. Few of these observers wrote specifically to record the natural history of these animals. Often, observations came from those who were commercially pursuing the bison or as anecdotes of people who incidentally encountered them. It has been the exhaustive task of the historical researcher to collect,

associate and distribute this information, often from obscure sources covering the breadth of the continent.

For the extensive period prior to European contact, archaeological and paleontological studies allow for the only direct means of studying animal populations (Larson *et al.* 2001; Cannon 2001). Skeletal remains provide physical data on prehistoric individuals and groups as sampled through specific mortality events. Traditionally, demographic information and individual biometrics can often be determined through the analysis of bone assemblages. These data, in conjunction with other contextual information, can help to discern the nature of the kill event which may be natural and somewhat random, or targeted, as in the case of archaeological assemblages. This "concrete" information is largely descriptive. Because the specific ecological context of prehistoric animals can only be inferred, developing ideas about prehistoric behaviours can be difficult. This is further complicated by the fact that assemblages represent only a sample of a prehistoric population (Frison 1991).

Modern, historic and prehistoric data on bison have previously been synthesized in attempts to better understand the natural history of the species throughout the Holocene in North America (Cannon 2001). The major problem with this approach has been the difficulty of using data from modern and historic sources to control for several indeterminate aspects of ecological context concerning prehistoric populations. The relevance of ecological models constructed in this fashion has been called into question. As a result, paleoecologists have increasingly turned to emerging techniques to derive contextual ecological information directly from prehistoric sources.

1.2 Archaeological Bone

Bone (including tooth and shell) is often the only directly organic remnant of long since dead animals. The analysis of faunal remains is a primary concern of modern archaeology. Techniques have expanded from the typical quantitative analysis of assemblages to a more qualitative approach including taphonomic and diagenetic studies (Behrensmeyer 1978; Lyman 1994), microwear and usewear (Kooyman 2000), and increasingly bone chemistry (Katzenberg 1992). Archaeological bone chemistry includes the study of both human and non-human remains and has grown to include

pursuits such as DNA extraction, analysis of lipids, trace element analyses, and stable isotope studies (Katzenberg and Harrison 1997, Tieszen *et al.* 1997b, Katzenberg and Saunders 2000).

Stable isotope analysis (SIA) has become an important tool for examining dietary relationships in both modern and ancient biological foodwebs. The isotopic composition of an organism's tissues, including bone, reflects the isotopic composition of its nutrient sources as ultimately dictated by the larger ecosystem within which these sources exist. This information can provide insight into the relationship between an organism and its environment. Since various organic elements move through an ecosystem using distinct natural cycles, the isotopic composition of different elements within an individual's tissues can yield information relevant to specific processes and nutrient sources.

1.3 Isotopic Reservoirs in Bone Tissue

Accurate and reliable chemical analysis of archaeologically recovered skeletal material is dependant upon the preservation of the chemical structure in bone which is deposited during the lifetime of the organism (Varney 1994). Qualitative alteration through diagenetic processes (physical weathering, chemical leaching and elemental replacement) during burial can be a major problem affecting the recovery of isotopic data from archaeological skeletal material (Lambert 1985). To correct for possible contamination, researchers will often "target" specific fractions of the sample bone via chemical extraction and subsequent purification (Ambrose 1993; Schoeninger *et al.* 1989).

Bone is composed of water and a primary collagen structure which supports a matrix of inorganic hydroxyapatite (Schwarcz and Schoeninger 1991). Dry bone is approximately 70% inorganic (hydroxyapatite) and 30% organic (collagen and non-collagen proteins) (Katzenberg 1992). Both organic and inorganic sources have been utilized in isotopic dietary studies and each presents its own benefits and limitations. Both carbonate and phosphate from the inorganic fraction have been studied as sources of dietary carbon. However, a consistent vulnerability of these fractions has been their tendency to exchange with carbonates and phosphates found in the ground water of the

burial environment (Schwarcz and Schoeninger 1991). Alternatively, collagen is relatively insoluble at the molecular level and even degraded bone may contain qualitatively intact collagen residues (Hall 1961; Schwarcz and Shoeninger 1991).

Collagen forms roughly 85-95% of the organic portion of bone (Katzenberg 1992), with the remainder composed of non-collagen proteins, fats and lipids (Shoeninger *et al.* 1989). Thus, collagen composes roughly 25% of dry bone mass. As a structural protein it performs many functions in the body, most of which require a tough, durable material and it is the principal organic component of skin, tendon, bone and dentine (Eastoe and Courts 1963). Collagen generally preserves well (Katzenberg 1992) and its isotopic composition reflects an integration of the dietary source of the animal (DeNiro and Epstein 1978, 1981b). Like all protein molecules, it contains carbon, oxygen, nitrogen and hydrogen; all of which are synthesized from an animal's various dietary inputs (Hall 1961). Because of the intimate structural relationship between collagen and hydroxyapatite, collagen may survive for a very long time depending upon the burial environment (Katzenberg 1992).

In practice, while the amount of collagen available in bone tissue may vary due to diagenetic alteration, the chemical nature of collagen tends to remain largely intact. Thus, while the physical preservation of bone may appear poor, the collagen that is present may still yield isotopic data that reflects antemortem values (Katzenberg 1992). Studies by Schoeninger *et al.* (1989), however, seem to indicate that extremely low collagen yields may be associated with qualitative changes in collagen content. Yields of 5% or lower have been found to be associated with aberrant stable isotope values. The exact reason for this correlation is still unknown (e.g. Schoeninger *et al.* 1989). Thus, collagen quality is of direct relevance to the determination of isotope values. Atomic carbon/nitrogen (C/N) ratios provide a quick, but widely employed method for verifying collagen integrity. C/N ratios of 2.9 – 3.6 are considered to be representative of collagen (DeNiro 1985). In addition, carbon and nitrogen concentrations within a sample should be considered. Ambrose (1990, 1993) suggests that only collagen with proportions greater than 3% carbon and 1% nitrogen should be analyzed. A more exacting, though somewhat more involved means of collagen verification, is Amino Acid Analysis in which the amino acid profile of a sample is compared to the "normal

profile" that is characteristic of the collagen protein (Ambrose 1993). Deviation from this norm suggests that contamination or degradation of the sample may have occurred (Katzenberg 1992).

Collagen is of further interest to archaeologists, biologists and ecologists because of its comparatively slow "turnover" in the tissues of mammals. In fact "collagen of the adult animal has so low a turnover value, that its half life is comparable in length to the period from maturity to death" (Hall 1961:32). Conservative estimates of collagen turnover are 10-20 years (Stenhouse and Baxter 1979). Thus, seasonal and other short-term variations average out and isotopic values in bone collagen represent an aggregate of an adult animal's dietary inputs over the better part of its life (Chisholm 1989).

1.4 Physical Properties of Stable Isotopes

Isotopes of a given element contain the same number of electrons and protons but differ by the number of neutrons in the nucleus. While different neutron quantities relate to differences in overall mass, these isotopes still possess the same charge balances and essential elemental "character" (Keegan 1989). Different isotopes of a particular element are known as "isotopic species" (Schwarcz and Schoeninger 1991). Variations in mass result in different reaction rates for elemental isotope species during a variety of physical and chemical processes and act to produce changes in the relative abundances of isotopes between the beginning and endpoints of a reaction (Shwarcz and Schoeninger 1991). These resultant changes are referred to as "isotope discrimination" or "fractionation" (DeNiro 1987; Ehleringer 1991).

While isotope discrimination may result from a variety of physical and chemical processes, it is the often regular and quantifiable discrimination that occurs between the tissues of an animal and its dietary sources that is of interest to archaeologists and paleobiologists. While radiogenic isotopes "decay", altering their relative proportions as a result of their own physical and electrical instability, stable isotopes discriminate through external mass-dependent processes (Keegan 1989; Schwarcz and Schoeninger 1991). As a result, stable isotope proportions in certain organic tissues (i.e. bone)

remain fixed even after the plant or animal dies (Keegan 1989). It is this stability that facilitates their use as dietary tracers.

Approximately 300 stable isotopes have been identified across all elements (Schoeninger and Moore 1992). Of these, isotopes of the five key light elements of the biosphere (C,H,N,O,S) are determined to be of primary biological interest (Schwarcz and Schoeninger 1991). Consequently, it is these "light" isotopes that are of importance to archaeological reconstructions of diet (Table 1.1). The central theme of stable isotope analysis of animal tissue is, essentially, "you are what you eat - isotopically". Put another way, tissue, if analyzed through the proper techniques, may yield dietary information for a particular individual. Measurements of isotope ratios have been used to reveal, among other things, physiological information for plants (O'Leary 1981, 1992; Lajtha and Marshall 1994) and animals (Tieszen *et al.* 1983; Ambrose 1993; Bryant and Froelich 1995), food web associations (Gearing 1991; Bocherens *et al.* 1995) climate (McKinnon 1986; Cormie *et al.* 1994c; Brooks-Lovvorn *et al.* 2001) and animal migration patterns (Chisholm *et al.* 1986; Hobson and Wassenaar 1997; Langemann 2000).

Elements commonly used in ecological studies have approximately 2 to 4 stable isotopes, with usually the lighter isotopic species significantly more abundant than the heavier (DeNiro 1987; Ehleringer and Rundel 1989). However, the reported isotopic ratio is actually the relative difference between the isotope content of the sample and a known standard gas (Boutton 1991a). Delta (δ) notation is used to express this difference. Thus, "$\delta^{13}C$ is the parts per thousand, or per mil (‰), difference between the ^{13}C content of the sample and that of the standard" (Boutton 1991a). Relative abundance of stable isotopes is measured using isotope-ratio mass spectrometry. This method was originally developed by Nier (1947) and modified several years later by McKinney and colleagues (McKinney *et al.* 1950). Conventionally, isotope ratios are written as δ values, using the relationship:

$$\delta X = (R_{sample}-R_{standard})/R_{standard} * 1000$$

Table 1.1 Average Natural Abundances (From Ehleringer and Rundel 1989), Applications and Substances (From Ambrose 1993) Analyzed For the Stable Isotopes of Major Elements of Interest in Dietary and Environmental Studies

Element	Stable Isotope	Percent Abundance	Applications	Substances
Hydrogen	^{1}H ^{2}H	99.985 0.015	Climate; plant water metabolism And photosynthetic mode	Water; cellulose; collagen; lipids; chitin.
Carbon	^{12}C ^{13}C	98.89 1.11	Animal diet, plant water use efficiency and photosynthetic mode; climate and habitat	All organic matter; shell and other carbonates; soil; bone collagen and carbonate; coal, gas, oil, water bicarbonate; atmospheric CO_2.
Nitrogen	^{14}N ^{15}N	99.63 0.37	Animal diet; plant and soil N-fixation; animal water use; climate; groundwater pollution	Animal, plant and soil organic matter; soil ammonium and nitrate; groundwater.
Oxygen	^{16}O ^{17}O ^{18}O	99.76 0.04 0.20	Climate; plant and animal water metabolism	Water; bone, soil and shell carbonate; bone and sediment phosphates; silicates.
Sulphur	^{32}S ^{33}S ^{34}S ^{36}S	95.00 0.76 4.22 0.02	Marine vs. terrestrial diet; air pollution	Organic matter; coal, oil; sulfates; sediments.

where X is the heavier isotope (e.g. ^{13}C, ^{15}N, ^{18}O, D), R_{sample} is the ratio of heavy to light isotopes in the sample (e.g. $^{13}C/^{12}C$, $^{15}N/^{14}N$, $^{18}O/^{16}O$, D/H), and $R_{standard}$ is the corresponding ratio of a standard (Ehleringer and Rundel 1989). Arbitrary standards for commonly measured elements have been universally accepted; for example, PeeDee Belemnite (PDB) is the standard for carbon, atmospheric nitrogen gas (AIR) for nitrogen, and Vienna standard mean ocean water (VSMOW) is the standard for oxygen and hydrogen (Ehleringer and Rundel 1989). Materials that have a higher relative abundance of the heavier isotope than the standard, have positive δ-values and are "enriched" relative to the standard. Materials having less of the heavier isotope have negative δ values and are "depleted" relative to the standard.

1.5 Sources of Isotopic Variation (Discrimination)

A variety of natural isotopic discrimination processes result in the formation of biologic, geologic and atmospheric "pools" with different isotopic ranges (Schwarcz and Schoeninger 1991). The origin of biological material can be elucidated based on differences among these isotopic pools in the environment. Basic discrimination processes that help to account for these variations include thermodynamic equilibrium effects and kinetic equilibrium effects (Ehleringer and Rundel 1989).

Completion of various chemical and physical reactions theoretically results in an isotopic molecular mass-balance between the product of a reaction and its initial substrate. In effect, they achieve equilibrium. What constitutes equilibrium for different substances, reaction processes and environments, is largely mediated by the mass dependant bonding properties of different elemental isotopes and temperature (Ehleringer and Rundel 1989, Ambrose 1993). Different isotopic species of the same element have slightly different thermodynamic properties. Typically, lower mass isotopes form weaker bonds within molecular substances (Hoefs 1973). Consequently they require less energy to motivate reaction and result in conditions for equilibrium that are temperature (energy) dependant (Hoefs 1973).

During reaction, heavy isotopes have a tendency to concentrate in the molecule with the bond having the greatest strength. Thus, at chemical equilibrium, the compound with stronger bond strength will be enriched in the heavier isotope relative to

compound(s) with weaker bond strength (Schoeninger and Moore 1992). The magnitude of these equilibrium isotope effects are also temperature dependent, producing less discrimination at higher temperatures (Hoefs 1973). This characteristic translates into the previously noted tendency for lower mass isotopes to have higher reaction rates than heavier ones (Hoefs 1973; Schwarcz and Schoeninger 1991).

These "kinetic" differences can result in differences in the relative abundances of elemental isotopes between a substrate and its end product during a reaction, depending on the duration of the reaction. Faster reacting, lower mass isotopes reach the reaction end point in less time than the heavier isotopes of an element (Keegan 1989). Lower activation energy and an enzymatic preference for "lighter isotope" molecules, discriminates compounds such that the products of a reaction are usually depleted in the heavier isotope relative to the reactants (Ehleringer and Rundel 1989). Thus, reactants and products reach a rate-dependent kinetic equilibrium in which the isotopic difference is constant. If, however, enough time is allowed to pass and the reaction moves to completion, no discrimination occurs and the final product will have the same isotopic ratio as the substrate, although isotopic equilibrium is seldom attained between organic molecules in biochemical systems (Schwarcz and Schoeninger 1991).

Rate-dependant kinetic equilibrium is predominant in biological systems (Schwarcz and Schoeninger 1991). Because the resulting discrimination is both constant and often predictable, source of origin may be revealed through isotopic analysis of biological and ecological material. By extension, isotopic analyses may also comment upon the specific chemical and physical processes by which discrimination has taken place such as photosynthesis, metabolism, and phase-change effects like evaporation and condensation (Hoefs 1973).

Isotopic systems of different elements may be better suited to examine specific processes or isotopic reservoirs. For example, temperature-mediated phase-change discrimination resulting from vapour pressure differences of isotopic compounds, causes changes in the isotopic composition of oxygen and hydrogen in meteoric waters (Hoefs 1973; Gat 1980). Thus, oxygen and hydrogen isotopic ratios may be used to trace climate in hydrologic systems. Most terrestrial isotopic variation in the carbon cycle occurs during incorporation into the biosphere during photosynthesis (Ehleringer

1991). Further discrimination occurs during metabolism when consumers ingest plant tissues and again when carnivores eat consumer tissue (Gearing 1991). Movement though the foodweb has a similar impact on the nitrogen isotope ratios in organisms (DeNiro and Epstein 1981b; Ambrose 1991). These consistent and quantifiable enrichments are known as "trophic-level" effects and are an important source of kinetic discrimination to researchers (Schoeninger and Moore 1992).

Finally, it is also important to remember that equilibrium and kinetic discrimination are both temperature-mediated. Thus, for most ecological and biological processes, the degree of discrimination will have at least some relationship to temperature and will influence the isotopic composition of the various geologic, biologic and atmospheric pools. This relationship may, however, be complicated by the interaction of other less directly temperature-mediated factors.

1.6 Stable Carbon Isotope Analysis

The use of stable isotope analysis in archaeological and ecological studies grew out of radiocarbon dating work in the 1960s (Keegan 1989). Researchers noticed that corn samples tended to yield dates that were younger than expected when compared to dates from other plant materials (Bender 1968). The subsequent discovery of different photosynthetic mechanisms within different plant species led to a realization that carbon isotope compositions may be used to distinguish plant species (Teeri and Stowe 1976) and animal diets (DeNiro and Epstein 1978). Thus, biological applications of stable isotope analysis began with the study of carbon isotopes and it is of little surprise that most archaeological research has focused on applications of carbon isotope analysis.

Differences in stable carbon isotope composition have been used to examine compositional differences between ecosystems, aspects of plant physiology (O'Leary 1981) and distribution (Teeri and Stowe 1976; Boutton *et al.* 1980), and animal diet (DeNiro and Epstein 1978; Hobson and Schwarcz 1986) and migration (Chisholm *et al.* 1986; Langemann 2000). Archaeological and paleoecological studies have addressed similar issues including the dependence of humans and animals upon marine or terrestrial resources (Chisholm *et al.* 1982) and maize consumption (van der Merwe and Vogel 1978). Climate has been examined by inferring past plant distributions from the

carbon isotope composition of soils (Steuter *et al.* 1990; Clark *et al.* 2001) as well as herbivore tissues (McKinnon 1986, 1990; Leyden and Oetelaar 2001; Brooks-Lovvorn *et al.* 2001).

Atmospheric CO_2 is the carbon source for terrestrial plants and is fairly uniform (about –7‰) although a slight decline in the atmospheric ^{13}C concentration (~-1.2‰) has been observed over the past century due to fossil fuel burning (Seuss effect) (Boutton 1991b; Tieszen 1994). Carbon enters the food chain when fixed into plant tissue carbohydrates during photosynthesis. This results in isotopic discrimination through the preferential incorporation of the light isotope (^{12}C) from atmospheric CO_2. The degree of discrimination between atmospheric CO_2 and plant tissue depends on the photosynthetic pathway used by the plant, in particular the enzymes involved in initial carboxylation (Ehleringer 1991). Consequently the $\delta^{13}C$ value of plant tissue is correlated with the mode of carbon fixation.

The Calvin cycle (C_3) photosynthetic pathway, used by most temperate and forest region plants, reduces CO_2 to a three-carbon compound (Boutton 1991b). Most of the temperate zone and all forest communities are dominated by C_3 species. These include cool-season temperate grasses and most plants used as food including wheat, rice, beans, tubers and nuts (Keegan 1989). The Hatch-Slack (C_4) pathway reduces CO_2 to a four-carbon compound is generally found in tropical and subtropical grasses, as well as some important cultigens like maize (Melbye 1984; Keegan 1989; Boutton 1991b). Many drought-resistant succulents (O'Leary 1981) use Crassulacean acid metabolism (CAM). Plants using the CAM photosynthetic pathway employ both C_3 and C_4 types of CO_2 fixation, the intensity of each being determined by environmental conditions. $\delta^{13}C$ values of CAM plants can be difficult to distinguish from those of C_3 and C_4 plants.

Stable carbon isotope analyses of herbivore tissue are primarily used to distinguish proportionate dietary inputs from amongst these plant groupings with distinct ranges of isotopic composition. Subsequent trophic isotope discrimination does occur when plant carbon is incorporated into consumer tissue (+5‰), and as carnivores consume other animals (+1‰); however, "most of the natural isotopic fractionation of interest to biologists results from carbon isotope fractionation during photosynthesis"

(Boutton 1991b). The isotope values for C_3 and C_4 plants exhibit a bimodal distribution in $\delta^{13}C$ with C_3 plants with mean values ranging between -26‰ and -28‰, while C_4 plants range between -12‰ and -14‰ (Tieszen 1994). Calculated averages are -26.5‰ for C_3 plants and -12.5‰ for C_4 plants (Chisholm *et al.* 1986; Tieszen *et al.* 1997b). Thus, the theoretical distribution of forage plant $\delta^{13}C$ values across the Great Plains is approximately 14‰.

Due to a 5‰ ^{13}C enrichment of consumer collagen relative to dietary forage, a herbivore diet consisting of 100% C_3 forage would be expected to produce $\delta^{13}C$ values in bone of -21.5‰ (-26.5‰ + 5‰). Conversely, an exclusively C_4 diet would produce mean $\delta^{13}C$ values of -7.5‰ (-12.5‰ + 5‰) (Chisholm *et al.* 1986). Dietary $\delta^{13}C$ values should therefore, range between -21.5‰ and -7.5‰, where a value of -7.5‰ would reflect a diet composed entirely of C_4 plant material, and a value of -21.5‰ would represent a diet devoid of C_4 plants. This spread in $\delta^{13}C$ value of approximately 14‰, is substantial enough to be reflected by consumers. Those feeding on both plant types would have intermediate tissue $\delta^{13}C$ values from which the relative proportion of C_3 and C_4 plants can be calculated using a simple linear interpolation (Chisholm *et al.* 1986). The following equation can be used to calculate approximate C_4 dietary contributions based on $\delta^{13}C$ values: (Schwarcz *et al.* 1985):

$$C_4\,\% = \frac{(\delta^{13}C_{\text{measured}} - \delta_3 - \Delta_{dc})}{\delta_4 - \delta_3} \times 100\%$$

where $\delta^{13}C_{\text{measured}}$ is the measured carbon isotope composition of an animal's bone collagen, δ_3 and δ_4 represent the average $\delta^{13}C$ values from within the range exhibited by most C_3 plants (-26.5‰) and most C_4 (-12.5‰) plants respectively, and Δ_{dc} represents the average trophic discrimination (5‰) between an animal's diet and its bone collagen (Tieszen 1991).

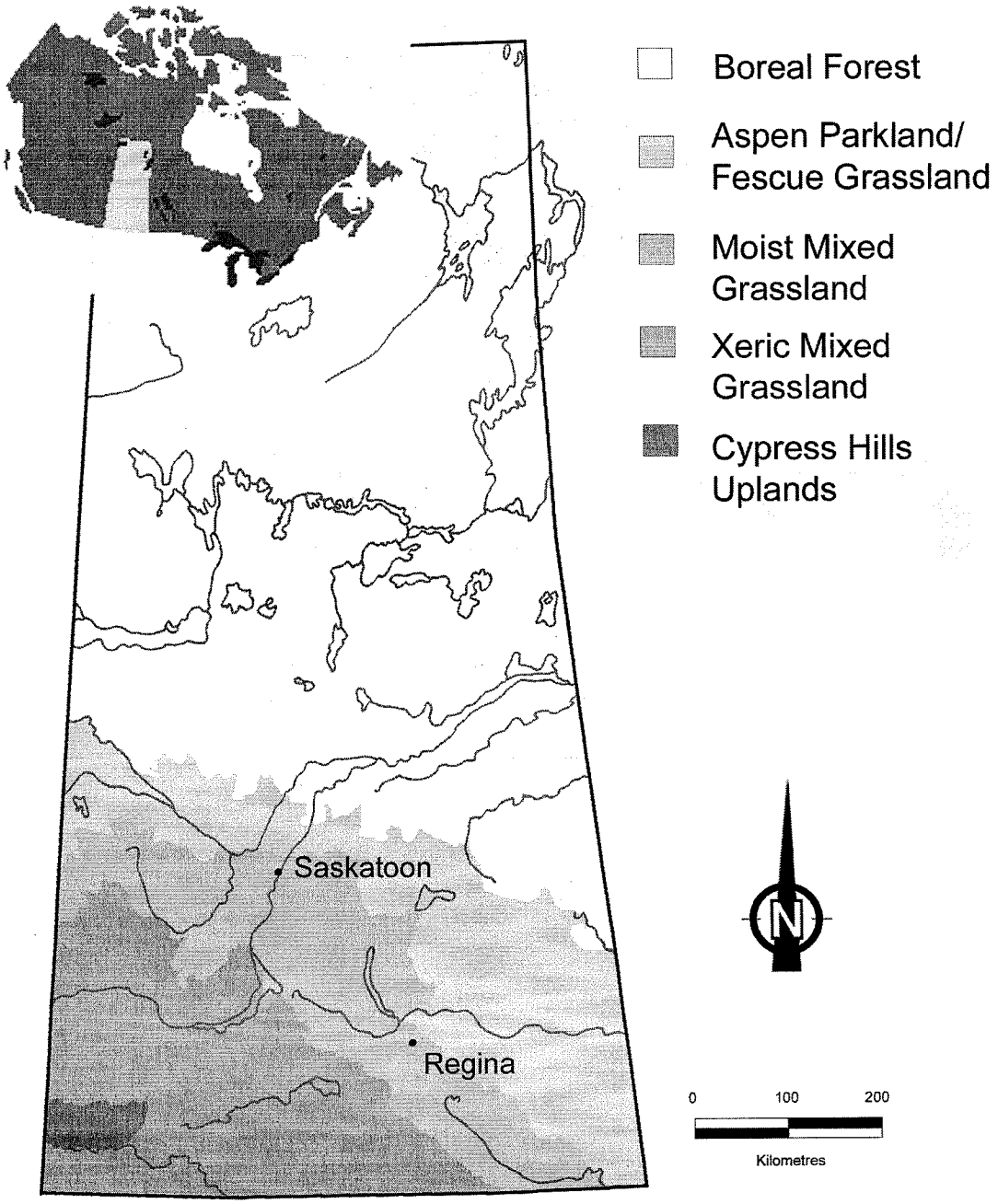
1.6.1 Terrestrial Plant Distributions

Although C_4 species occur primarily in tropical or subtropical grasslands, their northern limit in the Americas is defined as the Canadian grasslands (Chisholm *et al.* 1986). In southern Alberta and Saskatchewan, Coupland (1961) used the relative contribution of major plant species to define local vegetative communities. In Saskatchewan, these communities are distributed in bands from the south of the province towards the north (Figure 1.1). Morgan (1980) simplified this classification into three major vegetative zones including the Xeric Mixed Grassland (short grass), Moist Mixed Grassland (mid grasses) and the Fescue Grassland. In the Canadian Plains, the only C_4 grass found significantly in most vegetative communities and contributing to bison diet is *Bouteloua gracilis* (McKinnon 1986; Chisholm *et al.* 1986). According to Coupland (1961), *B. gracilis* (Blue Grama Grass) contributes negligibly to the Fescue Grassland, comprising only about 1.0% of overall forage production. This representation increases to a mean value near 14.5% in the Moist Mixed Grassland and ranges between 37.0% and 56.5% within the Xeric Mixed Grassland.

Looman (1983b) verified this trend proposing that the major differences in the distribution of grasses on both local and regional scales were related to variations in moisture regimes. At the local scale, he suggested that high temperatures and an initial increase in growing-season moisture would serve to stimulate the seedling-establishment and growth of *B. gracilis*, a drought tolerant species. However, subsequent success after initial establishment was at least partially dependant upon a drop in summer moisture whereby the drought-tolerant physiology of *B. gracilis* conferred an advantage. Thus, provided annual moisture is distributed adequately, an overall decrease in precipitation can be beneficial to the establishment of warm-season grasses. Actually, Coupland (1961) had already demonstrated that increasing moisture or temperature regimes could have a dramatic effect on the percentage of C_4 grasses. During the 1940s, annual rainfall was low in the Prairie Provinces, especially when compared to that of the early 1950s. As a result, the mean cover of *B. gracilis* in the Moist Mixed Grassland dropped from 17.5% in the 1940s to roughly 11.6% in the 1950s (Coupland 1961).

Distribution of C_3 and C_4 plants across the plains is highly correlated with variations in temperature and precipitation (Teeri and Stowe 1976; Boutton *et al.* 1980;

Figure 1.1 Ecoregion Map of Southern Saskatchewan



Looman 1983b; Laurenroth *et al.* 1999). More specifically, as regional temperatures increase and precipitation decreases, the relative proportion of C_4 plants comprising the local vegetative community will increase. Temperature and precipitation regimes vary across the Great Plains along both elevational and latitudinal gradients, as well as on a more regional basis. Generally, biomass of C_4 plants increases as latitude and elevation decrease. This trend is most apparent with changes in latitude. For example, Tieszen (1994) reported between 68 and 82 percent of ground cover as C_4 in southwestern and southern Texas, changing to roughly 35 percent as far north as South Dakota with decreasing composition into Canada. It is important to note, however, that while fixed phenomena such as latitude, elevation, soil composition, and topography are limiting factors, changes in C_3 and C_4 biomass are primarily functions of variable factors such as temperature and precipitation (McKinnon 1986).

1.7 Stable Nitrogen Isotope Analysis

DeNiro and Epstein (1981b) demonstrated that the isotopic composition of animal tissue nitrogen could be used to obtain information about an animal's diet if its potential food sources had different $\delta^{15}N$ values. Most subsequent archaeological research has focused upon using stable nitrogen isotope compositions of human and animal tissues to delineate dietary reliance on marine versus terrestrial resources (Schoeninger *et al.* 1983; Schoeninger and DeNiro 1984). Increasingly, however, attempts have been made to examine nitrogen isotope variations within terrestrial foodwebs as they relate to plant and animal physiology, animal diet, and climatic context (Ambrose 1991).

Nitrogen enters the terrestrial biome largely through the tissues of plants that incorporate or "fix" nitrogen from two major sources, the atmosphere and soils (Schoeninger and Moore 1992; Pate *et al.* 1998). Soil nitrogen is usually more enriched in ^{15}N than is atmospheric N_2 gas and thus, plants that rely on soil nitrogen should have more positive $\delta^{15}N$ values than those obtaining nitrogen from the atmosphere (Lajtha and Marshall 1994). Legumes, such as beans, rice and lentils are examples of plants that fix nitrogen from soils (Katzenberg 1992). Non-leguminous plants, including most

grasses, predominate in the northern Great Plains of North America and almost exclusively compose the dietary forage of local ungulate species.

Beyond variation that is generated primarily by differences in the physiological mechanisms through which plants incorporate nitrogen, many researchers have noted that climate may also have a significant effect on nitrogen values in soils, plant tissues, and animal tissues (Ambrose 1991). Heaton *et al.* (1986) demonstrated a climatic influence on nitrogen isotope ratios in South African mammals with data showing that $\delta^{15}N$ values were negatively correlated with annual rainfall. In addition, plants occurring on saline soils tended to have higher $\delta^{15}N$ values than those found in forested environments. Not surprisingly then, forest-dwelling animal species tend to have lower $\delta^{15}N$ values than do savanna species (Ambrose 1993). Assuming that the enrichment of ^{15}N relative to ^{14}N is as constant in herbivore tissues as has been previously reported (near 3‰), then these environmental differences should be reflected in the $\delta^{15}N$ values of their bone collagen (Ambrose 1993). In a comparison of East African herbivores from modern and prehistoric populations, Ambrose and DeNiro (1989) recorded differences in the $\delta^{15}N$ values of bone collagen and noted their relationship to reconstructions of environmental change. Their data suggest that shifts of between 2‰ to 3‰ over time in an area may reflect significant environmental differences.

The apparent relationship of $\delta^{15}N$ values in animal tissues to habitat aridity may result from physiological responses related to dietary and water stress. Following ingestion, nitrogen in food is processed for use in the body. During dietary synthesis, normal trophic discrimination results in tissue proteins that are enriched with ^{15}N relative to diet (Hobson *et al.* 1993). Correspondingly, waste products such as urea become depleted. However, many animals in arid environments, including bison, demonstrate physiological adaptations for water conservation (Fizet *et al.* 1995). As habitat moisture decreases, bodily water may be increasingly retained for tissue use. This will result in a higher relative proportion of urea in the total volume of urine excreted. Since the nitrogen content of urea is enriched in ^{14}N relative to diet, the excretion of this concentrated urine will result in a ^{14}N depletion of the animal's tissues and consequently, a more positive $\delta^{15}N$ tissue value. (Ambrose 1991).

Protein-stress, resulting from a low quality diet, may also cause an elevation of the $\delta^{15}N$ composition of tissues. Insufficient protein intake may result in the breakdown and reutilization of existing tissues in the body (Hobson *et al.* 1993). Since dietary synthesis initially causes a trophic enrichment of ^{15}N in animal tissues, re-cycling of tissue proteins may result in a further elevation of $\delta^{15}N$ values. This process may actually enhance the effects of water-stress as tissue re-cycling may compound the nitrogen enrichment resulting from concentrated urea excretion (Katzenberg 2000).

In summary then, several potential factors may impact the nitrogen isotope composition of an animal's tissues. These may include: mode of nitrogen fixation at the base of the food web and subsequent trophic enrichment at later levels, soil properties, total aridity, water and protein intake levels amongst consumers, as well as gastrointestinal bacteria. All may play a role in determining the final $\delta^{15}N$ value of an animal's tissues, and their interactions are quite complex (Schwarcz and Schoeninger 1991). However, by limiting a study to the analysis of variation within a particular animal species from a particular physiographic region, it is possible to remove potential causes of isotope discrimination from consideration.

Trophic shifts, other than those from weaning (Katzenberg 1988), do not contribute to variation within a herbivorous species, but rather between them. Similarly, the Northern Great Plains possesses a negligible proportion of leguminous plants, and bison typically do not consume these species. As a result, mode of nitrogen fixation is largely constant amongst forage plants contributing to their diet. Physiological adaptations will only affect inter-species nitrogen variation if there are physiological differences within members of a species separated by great distances and physical barriers, or as a result of progressive physiological adaptation over long periods of time. While these physiological adaptations may help to explain differences in $\delta^{15}N$ measurements between species, differences within a species exhibiting such a mechanism must ultimately derive from differences in the degree of dietary or water-stress affecting these animals and these differences are probably environmentally-dictated.

1.8 Paleoecological Applications of Stable Carbon and Nitrogen Isotope Analysis

The bulk of ecological research using stable isotopes has focused on the transfer of carbon amongst different isotopic "pools" in the environment. A large number of ecological studies exist which have utilized carbon isotope analyses. Thus, the following discussion focuses largely upon studies relevant to the ecological and archaeological analysis of bison. Unfortunately, stable nitrogen isotope analysis has received far less attention in bison studies (Tieszen 1994). As a result, it is only cursorily mentioned in the literature and is not deeply examined here.

In 1986, Chisholm *et al.* published an assessment of prehistoric bison foraging and movement patterns on the Canadian plains as determined from stable carbon isotope ratios in prehistoric bison bone. The study hypothesized "that if bison move through different territories which contain different foods, then these differences may be evident in bison diet" (Chisholm *et al.* 1986:193). More specifically, as bison bone tissue records a long-term dietary aggregate of food sources, carbon isotope ratios from bison bone would not necessarily reflect the C_3–C_4 plant composition of available forage in the locality from which these bison remains were recovered. This would indicate that these bison had acquired forage from a compositionally distinct region.

Chisholm *et al.* (1986) acquired several archaeological samples from Alberta, and a few from Saskatchewan, Manitoba, and British Columbia. Most of the kill sites used in the study came from the fescue prairies on the periphery of the Canadian grasslands. The study's only conclusive result was that the remains of these animals had yielded isotope ratios indicating higher dietary proportions of C_4 grasses than currently occur in the region. The proportions were high enough to suggest that the animals could not possibly have obtained all of their diet locally. They concluded that these bison must have moved deeper into the short-grass plains at some point in their yearly round (Chisholm *et al.* 1986). The study also outlined several factors which could contribute to variation in the utilization of C_4 plants by bison including: the annual and seasonal availability of C_4 plants, the seasonal movement of bison, differential assimilation of protein by bison in different seasons, and dietary preferences of the animals.

More recently, attempts have been made to assess the degree to which bison may have been resident in the peripheries of the plains. Archaeological bison bone from two different localities, Waterton Lakes National Park (WLNP) and Banff National

Park (BNP), provided loci for examining prehistoric bison from the inter-montaine, foothills regions on the western periphery of the Canadian plains (Varney *et al.* 1997; Langemann 2000). Of the 28 specimens examined by the study, three exhibited low proportions of C_4 grass in their diet (<8%), while 24 showed a significant C_4 contribution (10-28%), values which were comparable to a control sample from Milk River deep within the short-grass plains (20.7%) (Langemann 2000). It was concluded that the 24 animals with significant C_4 grass diet must have been at least partially resident in the western short-grass plains (Langemann 2000).

These studies were motivated by a desire to examine archaeological issues related to bison movement between the plains and peripheries (Kay and White 2001) and to explore issues surrounding the re-introduction of bison back into their former ranges within these national parks (White *et al.* 2001). Understanding the movement of prehistoric bison can have important practical implications, particularly from within regions where they no longer occur. Re-introduction of animals back into their former ranges necessitate the definition of what those former ranges were, the degree to which animals were resident in one area over another, and how predictable these movements may have been. All contribute to a better understanding of the impact that would result from reintroduction and its subsequent feasibility (White *et al.* 2001).

It has also been suggested that variations in the carbon isotopic composition of prehistoric animal tissues may reflect the association between the distribution of C_3 and C_4 plant species and climatic conditions. In 1986, McKinnon used stable carbon isotope ratios of prehistoric bison bone collagen from different time periods at a single location in Alberta, Head-Smashed-In-Buffalo-Jump (HSI), to examine differences in climate through time. Analysis of 44 individuals representing five broad time periods demonstrated $\delta^{13}C$ fluctuations over a range of 3.6‰ with some consistency amongst animals at each time period. A further study (McKinnon 1990) showed similar results for bison remains from the Cranford site 80km east of HSI. Here, 17 individuals representing five broad time categories over the last 4,500 years were analyzed. In both cases, it was assumed that modern bison, foraging in each region, would demonstrate bone collagen $\delta^{13}C$ values that reflected the C_3-C_4 grass composition locally. By comparing these theoretical estimations of modern C_4 diet composition with those

determined for archaeological bison at each time period, a qualitative assessment could be made about past plant distributions relative to modern ones.

Questions have been raised about the shortcomings of such an approach. Tieszen (1994) has suggested that it is difficult to know whether the excursions found by McKinnon (1986) are accurate reflections of changing climatic or vegetation patterns. Firstly, the fact that bison are mobile grazers suggests that "individual values could reflect a real climate change or, they could represent a sojourner from some different vegetation system" (Tieszen 1994:278). Secondly:

> "it is difficult to know whether the variation between sample dates is significantly different from that possible at one time. At present, it is unknown what the variation is in free-ranging bison at one point in time. A larger number of replicates from a short time interval (a few decades) are needed to derive an acceptable mean for that period" (Tieszen 1994:278-279).

American researchers have compiled $\delta^{13}C$ values for a large number of prehistoric bison from several archaeological sites in the American Plains which span the duration of the Holocene (Tieszen 1994; Tieszen *et al.* 1997b). A linear regression of over 200 bison demonstrated a distinct trend with an approximate 2.5‰ increase over time from the end of the Pleistocene to the present (Tieszen 1994). Nevertheless, an intra-site variability study showed that the variation within a population could be as great as ± 3 - 4‰ (Tieszen 1994). Controlled feeding studies of modern bison from Wind Cave National Park, South Dakota, raised from birth to death without supplemental feed, demonstrated little variability within the 19 individuals examined. The mean $\delta^{13}C$ value for the group was -18.7‰ ±0.2 (Tieszen 1994). However, since modern populations are often restricted in terms of movement and tend to be under less dietary stress, it has been suggested that archaeological populations may better represent the natural variation within a free-ranging herd (Tieszen 1994).

These limitations have had an impact on subsequent studies of carbon isotope variability within and between groups of archaeological bison. Jahren *et al.* (1998) analyzed eleven adult bison rib bones for carbon and oxygen isotope composition. The source of the bone was an early Holocene (9500 BP) Paleoindian bison kill site from Northwestern Nebraska known as the Hudson-Meng bonebed (Jahren *et al.* 1998). Both

collagen and carbonate were analyzed for carbon isotope composition and oxygen isotope composition was also determined for the carbonate fractions. It was noted that variation among the $\delta^{13}C$ and $\delta^{18}O$ carbonate values seemed to correlate with the location of each sample in the bonebed and consequently, three of the samples were discarded for concerns of possible contamination. While the remaining carbonate values suggested a diet averaging about -20‰, the collagen determined values ranged between -21‰ and -24‰. Despite the previously discussed concerns with the carbonate values, and while noting that the variation within the collagen values may be the result of a natural population, the authors decided to base most of their analysis on the carbonate data (Jahren *et al.* 1998). The small variation, seen amongst the remaining eight carbonate samples led to the conclusion that "this intensity of sampling was adequate to find an overwhelmingly central trend at this site" (Jahren *et al.* 1998:473).

The mean carbon values for both the carbonate and collagen fractions were found to be less negative than the $\delta^{13}C$ value of the modern local grass biomass. The authors suggested two potential explanations for these results. Firstly, "it is possible that the grass biomass available to grazers in the area at 9,500 BP had a higher $\delta^{13}C$ value, containing a larger % - mass of C_4 plant species"; and alternatively that the Hudson-Meng bison may have been "preferentially selecting a more C_4 plant rich diet than was characteristic of the environment" (Jahren *et al.* 1998:474). They also contend that the second hypothesis would have required that "bison migration and mobility patterns would have necessarily covered more land area when compared to modern bison" (Jahren *et al.* 1998:474).

Selection of one of these hypotheses as the "more probable reality" is confounded by a general lack of knowledge concerning the climatic and behavioural contexts in which prehistoric bison existed relative to modern animals. Interestingly, Jahren *et al.* (1998) note the potential of stable oxygen isotope ratios from bone carbonate, to yield data concerning the isotopic composition of an animal's ingested water and by extension, prevailing climatic conditions. The mean $\delta^{18}O$ ratio of the eight remaining specimens was approximately 21.9‰ with up to 1.5‰ variation (Jahren *et al.* 1998). Unfortunately, no further analysis of the oxygen data is offered.

More recent isotopic studies of bison remains have attempted deal with these contextual limitations in innovative ways. Leyden and Oetelaar (2001) analyzed bone collagen specimens of 11 individuals from four distinct time periods respectively dated to 9,000, 7,000, 5,000, and 2,000 years BP. All samples came from a single archaeological site in southern Alberta. While the number of samples for each time period was small (2-5), the standard deviation among both $\delta^{13}C$ and $\delta^{15}N$ values within each group ranged less than 1‰.The 7,000-year old group was distinct from the others on average, with $^{13}C/^{12}C$ ratios 1.5‰ more positive and $^{15}N/^{14}N$ ratios almost 2‰ more positive (Leyden and Oetelaar 2001). The authors compared the isotopic data to proxy paleoenvironmental data derived from the site itself and the larger physiographic region. Pollen cores, plant remains, animal bones, gastropods and soil organics suggest that the local and regional climates were considerably warmer and drier at this time period as compared to the other archaeological occupations and the current local environment (Leyden and Oetelaar 2001).

Acknowledging the limitations of drawing inferences from small sample sizes, the authors conclude that bison from the 7,000-year old sample group were "either foraging in an environment that was warmer and drier than modern times, or exhibiting a foraging strategy that was different from that expected in modern herds" (Leyden and Oetelaar 2001). Given the previously noted reconstructed environmental context from 7,000-years ago, it is suggested that the positive shifts seen in both the $\delta^{13}C$ and $\delta^{15}N$ values in bones from this time period may be linked to a warming and/or drying climate. It is cautioned however, that such interpretations may only be accurate in simple ecological contexts where the climatic shifts are known to be significant and sustained (Leyden and Oetelaar 2001).

Brooks-Lovvorn *et al.* (2001), take this approach one step further by linking manifestations of Paleoindian culture to paleoclimate changes as determined through the stable carbon isotope composition of bison bone from several prehistoric sites. Using calculated C_4 dietary compositions, the authors inferred changes through time in forage distributions for each of five separate time periods sampled in Northwestern Wyoming. Data concerning annual and seasonal temperature and precipitation, as related to the distribution of C_3 and C_4 plants in the modern local environment, were

used to create predictive models. Linear regressions developed from these data allowed for the calculation of precipitation and temperature for each of the archaeological time periods.

Comparison of the modern data to the calculated past data led to several conclusions. Firstly, the period between 12,400 to 11,900 BP appeared to be cooler and moister than present, although by its end, it had begun to rebound. Second, the occupation dated to 7,300-years BP, indicated a hotter, wetter climate than present. The authors linked the early climate episode to the apparent split of Early Prehistoric peoples at the end of the Pleistocene into separate but concurrent, plains and mountain-foothills cultures (Brooks-Lovvorn *et al.* 2001). The latter climate period was linked to the hypothesized era of plains "abandonment" during the Mid-Holocene climatic optimum. Despite the conventional view that hot, arid conditions primarily motivated reduced habitation on the plains at this time, Brooks-Lovvorn *et al.* (2001) suggest that hot, wet conditions may have been evidence of sudden changes in overall atmospheric circulation patterns. This would have resulted in a highly variable climate with concomitant periods of aridity and near monsoon rain.

Concerns must be addressed about some of the assumptions in this study. Increases in the C_4 portion of bison diet may not necessarily be directly translatable into estimates of forage distribution. Although bison are relatively unselective grazers, their subsistence strategies may vary in time and space (Bamforth 1988; Frison 1991; Hart 2001). In addition, interpretation of the Mid-Holocene climatic optimum is usually as a xeric (hot/dry) period of variable duration potentially manifesting at different times on different parts of the Great Plains (Antevs 1955; Vance *et al.* 1995; Clark *et al.* 2001).

1.9 Stable Hydrogen Isotope Analysis

Hydrogen isotopic compositions of plant and animal tissues have primarily been used to examine parts of the hydrologic cycle. While measurements of δD have been used quite extensively by plant physiologists (Sternberg 1989) their application involving consumers has been limited. Animal tissue studies have demonstrated potential in several areas including: as natural biomarkers in migratory birds (Chamberlain *et al.* 1997; Hobson and Wassenaar 1997) and butterflies (Wassenaar and

Hobson 1998), and to infer temperature and relative humidity of paleoclimates from insect chitin (Schimmelmann and DeNiro 1986; Miller *et al.* 1988) and mammal tissues (Cormie *et al.* 1994b, 1994c). Theoretically, isotope ratios of oxygen and hydrogen comment upon the same ecological processes in their capacity as formative components of water (H_2O, HDO), although specific differences in their physical properties do provide for some differences in chemical behaviour. Stable isotope ratios of hydrogen and oxygen in precipitation are linearly correlated (known as the "meteoric water line", MWL) (Ehleringer and Dawson 1992).

The isotopic composition of atmospheric waters is primarily a function of temperature change as air masses move over the continental landscape. Generally speaking, the further away water vapour travels from its source (i.e. the ocean), the more negative its isotopic values become. This depletion is controlled by at least three straightforward factors including altitude, distance from the coast, and latitude. For the most part, an increase in any of these variables results in a corresponding isotopic depletion in the deuterium concentration of atmospheric vapour (Gat 1996). These effects are temperature-dependant and relate to cooling of the air mass by each spatial characteristic.

The mechanism for this depletion is the discrimination related to vapour pressure differences causing D and ^{18}O to favour the liquid rather than vapour phase, or solid rather than liquid phase (Gat 1996). As water evaporates from the ocean surface, water molecules containing the heavier isotopes are discriminated against causing the vapour to be depleted. As clouds move inland, that water vapour becomes even more depleted as D and ^{18}O are preferentially lost through condensation and subsequent precipitation (Clark and Fritz 1997). Further precipitation is progressively more depleted and, therefore, inland bodies of freshwater that receive their major input from meteoric water are generally more depleted than bodies closer to the coast (i.e. closer to the original vapour source). The North American continental distribution of deuterium isotopes is a marked gradient of positive values in the southeast towards negative values in the northwest.

The degree of isotope discrimination is also temperature-dependent, being more pronounced at lower temperatures (Gat 1996). In cooler areas and in the continental

interior, discrimination occurs to a greater degree. Thus at mid- and high-latitudes, even small temperature changes can yield significant isotopic differences in the composition of atmospheric water vapour. Temperature is therefore the primary factor mediating isotopic composition in meteoric waters from these locations. This process dominates as long as the ocean is the major source of moisture and there is relatively little input from re-evaporated moisture. At lower latitudes and in coastal areas, where the climate is warmer, deuterium content of precipitation is correlated with volume of rainfall (Schotterer *et al.* 1996). Proximity to the ocean or large bodies of water also promotes re-cycling of water vapour through repetitious evaporation and precipitation. In addition, increased surface humidity and stronger convection in warmer and coastal climes promotes mixing of air masses and may result in isotopic exchange (Gat 1996). These phenomena thus act to mediate or even mask simpler temperature-dependant processes.

The stable isotopic composition of groundwater will depend on the origin of the precipitation, the climatic conditions of the recharge environment, and the recharge process. However, in most hydrological settings the isotopic composition of the groundwater will closely reflect the isotopic composition of precipitation (Fritz *et al.* 1987). The major engine of isotope discrimination in soil waters of the continental interior is evaporation. Increases in temperature between precipitation events may increase the rate of surface evaporation from soils. This acts to enrich soil waters with the heavier isotopic species (Gat 1996).

Hydrogen and oxygen from soil water enters the food web when fixed by plants. Therefore, the isotopic composition of plant tissues is controlled by the isotopic content of the water source (Ziegler 1989), and hence reflects the continent-wide isotopic gradients observed in precipitation (Yapp and Epstein 1982; Cormie *et al.* 1994a, 1994c). Water in plant leaves is isotopically enriched relative to ground water through evaporative transpiration. This enrichment is most intense in arid climates (Dongmann *et al.* 1974).

Significant isotope discrimination occurs during certain biological processes, such as photosynthesis, and may be dependent on enzymes in specific photosynthetic pathways. Characteristic isotopic differences among plants, based on the type of

photosynthetic pathways used, exist for hydrogen as they do for carbon. However, unlike carbon, hydrogen isotopic differences between C_3 and C_4 plants are small and cannot be used to distinguish photosynthetic pathways. Plants using the CAM photosynthetic pathway, however, are roughly 100‰ more enriched in deuterium than C_3 or C_4 plants (Sternberg 1989). Interestingly, the oxygen isotope composition of plant waters does not seem to demonstrate significant differentiation relative to the photosynthetic pathway used (Sternberg 1989). In addition to enzymatic and climatic effects on plant δD values, discrimination can occur among biochemical constituents of plant tissues. Different parts of the same plant can vary in terms of isotopic composition (Estep and Hoering 1980; Ziegler 1989; Smith and Epstein 1970).

Identifying the specific reservoirs from which water is incorporated into the tissues of consumers has generated debate amongst hydrologists and ecologists. While most researchers agree that body water is the internal source from which the oxygen and hydrogen isotopic composition of animal tissues are derived (Longinelli 1983; Luz *et al.* 1984; Koch *et al.* 1994; Cormie 1991; Cormie *et al.* 1994b), there is continuing debate over which environmental sources contribute to body water composition (Luz *et al.* 1990; Koch *et al.* 1994; Hobson *et al.* 1999). While some speculate that leaf water may be the dominant source of input water (Cormie 1991; Cormie *et al.* 1994b), most agree that body water isotopic composition is a synthesis of more than one environmental flux. Dietary water (leaf water) and drinking water likely both play an important, if not somewhat variable role in determining body water isotopic ratios (Hobson *et al.* 1999). Oxygen values may be further influenced by respiration of atmospheric oxygen gas (Luz *et al.* 1984; Koch *et al.* 1994).

Cormie *et al.* (1994c) argued that δD values in tissues of long-lived herbivores provide a time-integrated measure of plant tissue δD values in a region over the lifetime of the animal and, therefore, are good indicators of average growing-season precipitation δD values. While many studies have demonstrated the potential of δD values in organic material to predict temperature variations (Northfelt *et al.* 1981; Miller 1984; Cormie *et al.* 1994c; White *et al.* 1994; see White 1989 for review), investigators have yet to make inferences about unknown climates of the past using this method.

1.9.1 Hydrogen Exchange

The limited use of stable hydrogen isotope analysis is possibly due to the complication of hydrogen exchangeability and a more labour-intensive analysis (Wassenaar and Hobson 2000). Polar, covalent bonds between nitrogen or oxygen and hydrogen create strong dipole-dipole interactions that allow hydrogen atoms to exchange with each other, or with hydrogen from atmospheric water vapour. The presence of exchangeable hydrogen in a sample will alter its δD value. The D/H ratios of substances are partly dependant on the D/H ratio of the last water with which it came into contact (Grinsted and Wilson 1979).

Carbon-hydrogen bonds, on the other hand, are non-polar and there are no unshared electrons. As a result, hydrogen ions bound to carbon do not form dipole-dipole hydrogen bonds and are therefore, non-exchangeable (Cormie 1991). Samples that have been exposed to atmospheric water of different δD value or to atmospheric water for different lengths of time, for example in the laboratory or during storage, will take on a portion of the atmospheric D/H composition. Consequently, the overall, measured δD value of a material that has exchangeable hydrogen includes a portion that has been derived from atmospheric water vapour and does not truly represent the ecology of the animal. It is therefore important to account for this hydrogen exchangeability during sample preparation.

One method that has been developed for analyzing complex biological material involves standardizing labile hydrogen with water of known δD composition, effectively replacing exchangeable hydrogen with hydrogen of a known δD value (Grinsted and Wilson 1979; Schimmelmann 1991; Schimmelmann *et al.* 1993; Feng *et al.* 1993; Wassenaar and Hobson 2000). If the proportion of hydrogen that exchanges is also known, then the fixed, carbon-bound δD value can theoretically be calculated from the overall measured δD value. It has been suggested that exchangeable hydrogen in most biological tissues, including bone, should represent about ~20% of the total (Schoeller *et al.* 1986).

1.10 Paleoecological Applications of Stable Hydrogen Isotope Analysis

An important characteristic of the geochemistry of stable hydrogen isotopes is that hydrogen shows by far, the largest relative mass differences between its two stable isotopes (H with one neutron and D with two neutrons). Therefore, isotope discrimination effects can be particularly strong for hydrogen resulting in a great range in isotopic ratios amongst natural reservoirs (Hoefs 1973). The use of δD measurements in food web or nutrient-tracing studies has strong potential because of predictable isotopic differences among locations and biomes. It has been established that δD in precipitation follows a gradient of decreasing deuterium content from southeast to northwest on the North American continent (Hobson and Wassenaar 1997). Because δD values in precipitation are related to temperature (Gat 1996), and plants and consumers reflect local precipitation δD values (Yapp and Epstein 1982; Cormie *et al.* 1994c; Chamberlain *et al.* 1997; Hobson and Wassenaar 1997), investigators have suggested the use of δD values in plant tissues (White *et al.* 1994) and consumer tissues (Miller 1984; Cormie *et al.* 1994c) to reconstruct temperature changes in recent and geologic time.

Very few archaeological studies have attempted to analyze the biogeochemistry of stable hydrogen isotopes within the tissues of prehistoric fauna. Perhaps the most important such investigation was that of Cormie and colleagues (Cormie 1991; Cormie *et al.* 1994a, 1994c) in which the isotopic composition of modern white-tailed deer bone was examined from sites across North America. Theorizing that the isotopic composition of deer bone collagen would be primarily synthesized from the leaf water of dietary plants, the authors set out to reconstruct known temperature and relative humidity curves for a variety of sampling locations. It was experimentally-determined that approximately 20.5% of the total hydrogen atoms in terrestrial mammal whole bone samples were exchangeable (Cormie 1991; Cormie *et al.* 1994b). This value was derived from samples of southern Alberta fossil bison (1,500 BP) and modern musk ox bone. By applying these correction factors and taking into account relative humidity, accurate determinations of δD from modern deer bone collagen were made. A gradient of less depleted (~ -20‰) to more depleted (~ -100‰) δD values, running from the southeast (Florida) to the northwest (central Alberta) of the continent was noted. It was subsequently concluded that a significant direct relationship exists between the D/H

ratio of non-exchangeable hydrogen in deer bone collagen and that of local growing season rain δD (Cormie 1991, Cormie *et al.* 1994c). It is interesting to note that δD measurements were also obtained from prehistoric samples of bison bone collagen. Six samples from central Alberta (1,500 BP) averaged ratios of about -71‰, while two samples from the Yukon (12,000BP) averaged about -134‰ (Cormie 1991).

One other preliminary study from the United Kingdom, has examined non-exchangeable δD values from bone collagen of modern British mammals, birds and fish (Birchall *et al.* 2002). Nineteen different animal species were analyzed for the deuterium isotopic content of their bone collagen. The authors found that a general isotopic divide of about 30‰ existed between carnivores and herbivores. Nevertheless, a simple linear relationship between trophic position and D/H ratio was not evident. The complete range of δD values obtained for the study was between -71‰ and +144‰. Standard deviations of up to 10‰ were not uncommon for groups of a particular species (Birchall *et al.* 2002).

In an attempt to determine whether these analyses would be applicable to archaeological materials, the authors compared D/H ratios of archaeological (Iron Age) red deer bone collagen (n=5) to those of modern deer (n=94). The percentage of exchangeable hydrogen was determined to be 27.0 ±1.47% for archaeological deer and 24.8 ±6.25% for modern deer. Mean δD values for the archaeological deer were -11.6 ±6.98‰ and those of modern specimens were -24.0 ±6.76‰. The authors attributed these temporal differences to several possible causes including diagenetic alteration of the archaeological bone, differences in the δD of precipitation between the two time periods, and potential differences in diet (Birchall *et al.* 2002). It should be noted that geographic differences between temporally distinct sample groups would have less effect upon δD values in Britain, than in North America, as the island has a small variation in precipitation δD as compared to larger land masses. Consequently, no direct relationship can be drawn between the isotopic compositions of the tissues of individuals of the same species occurring on both continents.

1.11 Bison Ecology

To generate meaningful inferences, archaeologically derived data concerning the ecology of past bison must be interpreted through comparison to modern and historic sources. There are no extant "free-ranging" bison herds and all modern analogies must be considered within this context. Historic data can supplement modern interpretations by providing a glimpse into the ecology of individual animals and herds around the time of European contact, prior to the decimation of indigenous populations. Syntheses such as these represent the best approach to the reconstruction of recent, Late Holocene bison movement, aggregation and foraging patterns.

While modern bison are constrained in terms of movement, and herd sizes are far smaller than recorded in historic times, generalizations have been made about their behaviour. Modern bison tend to be relatively unselective grazers in comparison to highly selective herbivores such as antelope (Chisholm *et al.* 1986; Tieszen 1991). This characteristic may reflect the biological constraints of the animal. Although they can tolerate a relatively low-quality diet, bison require fairly large absolute volumes of forage due to their size (Peden 1976). It has been suggested that this generalist "lawn-mower" foraging model is too simplistic in many ecological contexts and has only been successfully applied to short-grass prairie settings (Larson *et al.* 2001). However, it is the assumption of this behavioural tendency in bison that allows for the analysis of conditions which encourage deviation from it.

If bison exhibit a "generalist-foraging" approach in short-grass prairie settings, it is probably because plant distributions in this biome encourage such a response. Modern plains bison appear to be well adapted to the grasslands and consume grasses almost exclusively (Peden *et al.* 1974; Bamforth 1988; Steuter *et al.* 1995). Nevertheless, bison will "tend to select the most nutritious forage available at any given time, consuming cool-season grasses when they are most nutritious during the spring and fall and switching to warm-season grasses during the summer when these species are at their most active periods of growth" (Bamforth 1988:79; also see Vinton *et al.* 1993; Steuter *et al.* 1995).

Bison do exhibit changing seasonal concentrations with respect to their consumption of C_3 versus C_4 forage. Isotopic values in the feces of naturally grazing bison reflect this seasonal disparity (Tieszen 1994). Tieszen (1994) records a 45%

increased consumption of C_4 forage during the late summer amongst a herd of un-managed South Dakota bison. Thus, the selective tendencies of bison seem to generally reflect the seasonal cycle of grassland forage. Vinton *et al.* (1993), note that in addition, while bison will preferentially forage for warm-season grasses in the spring/summer, they will also tend to select areas recently burned over others. These preferences likely result from increased grass production and the removal of dead plant tissue in recently burned areas (Vinton *et al.* 1993). In the autumn and winter, bison increased use of cool-season grasses without regard to whether an area had been burned or not. Burning did not dramatically tend to increase cool-season plant production, and thus, burned and unburned areas were grazed more uniformly (Vinton *et al.* 1993). It would seem that nutritional value, whether a result of seasonality or other processes, is the prime determinant of bison forage selection.

Several factors act to complicate this adaptation. Ungulates move around as much as required in order to obtain adequate food, water and shelter. Part of bison adaptation to plains environments reflects a generalized versatility. Regional distributions of forage and water, as influenced by weather, fire and previous grazing, largely determine the density of animals in an area (Hart 2001). These factors also control the size and boundaries of the areas in which they migrate, the size of the herds they move in, their overall degree of mobility and their specific migration patterns at any given time. Rainfall and temperature largely determine forage distributions. In addition, inter- and intra-species competition, especially as animal biomass within an area reaches carrying capacity, can motivate changes in range and forage selection (Huebner 1991).

A 21-year study of inter-species relationships in Yellowstone National Park monitored several animal populations that were allowed to forage and breed naturally with few human controls (Singer and Norland 1994). Detailed monitoring occurred at the beginning (1967-1970) of the 21-year study and towards its end (1986-1988). Between these two periods of time, the bison population increased 700% and the park's occupied range increased by about 300%. Bison diet also changed and reliance on grasses similarly increased. This likely resulted from several inter-related factors. First, a milder climate for period 2 produced less snow in the winter permitting larger winter

ranges. Second, mild climate combined with an increase in ungulate grazing likely stimulated grassland production and contributed to increasing protein content in available grasses. Finally, increases in intra-specific competition likely provided some impetus for range expansion (Singer and Norland 1994). Interestingly, while other ungulate species (elk, pronghorn, mule deer) also increased in both population and range, the unchecked growth of the park's bison herd suggests that interspecies pressures were negligible (Singer and Norland 1994).

The complexity of bison behaviour has made consensus virtually impossible. Any interpretations of prehistoric bison ecology have looked to modern analogues and historical sources to provide a generalized guide of how modern bison behave on the landscape. Significant debate has been centered on this point. As Bamforth (1988:80) stated:

> "Bison migration and aggregation patterns have been the subject of most of the anthropological debate over bison ecology, and the essence of this debate is tied to the question of how predictable these patterns actually were. As plains anthropologists have used the term, *predictability* seems to mean the degree to which herds of the same size returned to the same point on the ground at the same time of the year in successive years."

Bamforth (1988) suggests that these aggregation and migration patterns may be quite predictable if one first recognizes the factors determining these patterns (Bamforth 1988). Descriptions of historical and ecological observations have suggested that bison on the Canadian Plains may have experienced a fairly regular pattern of seasonal migration (Roe 1951; Moodie and Ray 1976). As described by Morgan (1980), this would entail wintering within aspen parklands with movement out onto the prairie grasslands in the spring and summer months. In contrast, Hanson (1984) argued that bison did not undergo regular seasonal movements, but instead demonstrated erratic patterns that were flexible and localized. Hanson (1984) based his reconstruction largely on an ecological analysis of bison in South Dakota which suggested that adequate forage existed year round to sustain resident herds. Several historical accounts dispute the nature of bison seasonal migration, suggesting that the movement either did not

occur or involved short, sporadic seasonal dispersion (Arthur *et al.* 1975; Garretson 1938; McHugh 1972).

Using data on the migration patterns of several African ungulates and North American caribou, Epp (1988) proposed a model wherein the bulk of the population migrated seasonally to and from the grasslands, while smaller resident populations remained throughout the foraging range. According to Bamforth (1988), the apparent differences between the Canadian herds and their southern counterparts were both understandable and predictable, given the different ecological contexts. Despite the regularity of movement in Canadian herds, data compiled by Moodie and Ray (1976) suggest that during milder winters when forage would not have been deeply buried by snow and cold and wind would have been less severe, migratory behaviour was reduced (Bamforth 1988). Therefore, the erratic and dispersed patterns observed by Hanson (1984) in South Dakota herds and depicted by some historical sources, are consistent with observations of Canadian bison in less severe winters.

In general then, bison behaviour may best be explained through the application of a model of "ecological determinism" (Trigger 1989). Effectively, modern plains bison are a species adapted to the grasslands of North America. Within this context, they are extremely versatile and have developed behavioural mechanisms to allow them to maximize the beneficial changes in their environments, and minimize the negative ones. Similarly, changing climatic variables, which alter forage and water distributions, cause or alleviate conditions of stress upon individuals or herds.

The intensity of this stress is determined by the degree of climate pressure combined with aspects of herd structure, density and regional topography (Huebner 1991; Hart 2001). Thus, decreases in forage and water abundance increase the tendancy of animals to move about in search of sustenance. Similarly, it reinforces their tendency to select the most nutritious forage available. The degree to which they are able to do this is dependant on topography, climate (in extreme cases), herd composition, and overall bison population density within a region (Singer and Norland 1995; Hart 2001). However, while there are seasonal cycles to climate across the plains, intensity and duration also change along latitudinal and altitudinal gradients. Along these same transects, it is reasonable to assume that differences will exist in the responses of bison

to seasonal changes and more generalized climate change. Thus, the degree to which bison are predictable is dependant upon the ability of people to assess these complex interactions. This may be easier for a specific region than for the entire Great Plains.

A further complicating factor is the continuous evolution of the species over the last 10,000 years. "We may be on somewhat dangerous ground in assuming that the extinct variants of bison demonstrate identical or similar behavioural patterns to the modern form" (Frison 1991:273). It has been suggested that evidence for differences between bison populations throughout the Holocene comes from the identification of morphological change in bison remains through time (McDonald 1981; Guthrie 1966, 1970; Wilson 1978; Bamforth 1988). In such a scheme, changes in bison morphology have been assumed to be related to changes in bison behavior in the context of the larger environment.

Progenitors of modern bison have existed in North America for at least 200,000 years. Bison as a genus are considered to have originated during the upper Pliocene in eastern Asia (McDonald 1981). A bizarre variety of forms have been described and many authors have postulated an incredibly complex taxonomic scheme involving as many as 20 to 25 distinct fossil forms (Arthur *et al.* 1975). Bison, like humans, apparently used the Bering Land Bridge to cross over into Alaska, although at a much earlier time (Wilson 1994). Throughout the Pleistocene, the area known as Beringia (Northern Siberia, Central Alaska/Yukon and the Bering Land Bridge), remained largely ice free, even at the heights of glaciation (Arthur *et al.* 1975).

The colonization of North America, by bison, was likely not characterized by a progressive linear evolution of the genus, but rather, involved an admixture of *in situ* evolution, genetic mixing as a result successive migrations, and the co-existence of multiple evolutionary lines (Guthrie 1970; Wilson 1994). Consequently, the evolutionary taxonomy of the genus is still largely speculative. Holocene changes in the size of bison have been considered to have resulted from pressures endemic to North American populations. Unfortunately, both Holocene bison taxonomy and the specific nature of this size change, are still highly debated (Helgason 1987). Despite evidence suggesting a tendency towards limited, seasonal migratory behavior in northern plains

bison during the later Holocene, it remains unclear as to whether this foraging strategy was used by earlier Holocene forms.

Forests, woodlands, savanna and steppe habitats covered much of Eurasia, Beringia and North America during the Pleistocene (McDonald 1981). Pleistocene savanna and steppe habitats were smaller and characterized by lower carrying capacities than modern mid-latitude grasslands. Pre-Holocene arctic steppe and tundra fauna was diverse (a profusion of Pleistocene megafauna) and inter-specific competition was probably high. Thus, inter-specific competition and a varied food base motivated a proliferation of species adapted to niche foraging, and yet kept overall numbers within a niche species in check. In such a context, favorable large herbivore traits would include adaptability to browsing or grazing dependant on conditions, and sufficient size and/or specific morphology to convey competitiveness (McDonald 1981).

The great number of Pleistocene forms has thus, been largely attributed to multiple lines of migration which continued to adapt, *in situ*, to specific environmental niches. This situation was largely fostered by the complex character of Pleistocene environments in North America which allowed for an increased diversity of forms, species and taxa, but which limited the carrying capacity within each niche group. In such an environment significant differences existed between contemporary bison taxa. *Bison latifrons*, the largest bison form ever to exist, occurred in mid-latitude North America, and at times, synonymously with *Bison antiquus*, the probable progenitor of the modern plains bison (McDonald 1981; Frison 1991). *B. latifrons* was characterized by large size of both body and horns. It has been assumed that this adaptation was favourable for competition. Additionally, the morphology of the occipitals and forward orientation of orbits suggests that the animal held its head higher than other bison forms and may have engaged in eye-level browsing (McDonald 1981).

If *B. latifrons* is seen as a forest/woodland form, then *B. antiquus* likely represented a smaller and stockier savanna-scrub variety. The key to this interpretation is the assumed primary range for *B. antiquus* during much of the Pleistocene. Most of the early specimens come from the Southwestern U.S. and Northern Mexico. Throughout much of the Pleistocene, this area was covered by desert scrub, and savanna-grasslands. In the late Pleistocene and early Holocene, the species range seems

to have shifted north and it has been widely assumed that *B. antiquus* opportunistically extended its range after the extinction of *B. latifrons* and other related forms. In addition, a potential subspecies of *B. antiquus* appears during Pleistocene/Holocene boundary times in the western Great Plains and by the early Holocene these two forms seem to exist as regional variants: *B. antiquus* in the south and *B. occidentalis* in the north.

By the end of the Pleistocene, these two variants remain as the only surviving bison forms. As such, both were still essentially animals of the Pleistocene savanna-steppe. Savannas and wooded steppes are structurally differentiated from primary grasslands such that the patterns of woody and herbaceous vegetation result in a habitat of great structural diversity. In contrast, North American mid-latitude grasslands, which expanded dramatically during the Holocene and are normally the result of both climatic factors and fire, are structurally simple and offer an abundance of space and continuously distributed herbaceous vegetation readily accessible to large herbivores. Grazing would be the only feeding strategy capable of supporting a viable population of large herbivores (Heckathorn *et al.* 1999). In such a situation, assuming that fewer species would adapt to the structurally simple grassland environment, intra-specific competition would be more important than inter-specific competition, especially as the bison population increased. High social cohesiveness and tolerance would be desirable (McDonald 1981).

Bison bison is morphologically differentiated from both *B. antiquus* and *B. occidentalis*. Modern plains bison are smaller than their early Holocene counterparts. In fact, "size diminution of bison has been a gradual process throughout the entire Holocene" (Frison 1991:272). Tip-to-tip spread of bison horn cores has decreased during the period at a rate of about 32mm/1000 years (Wilson 1978). Additional changes in skeletal morphology seem to continue along the lines of the suite of adaptations which differentiate *B. antiquus* from other forms like *B. latifrons*. These include the downward rotation of the head, lateral placement of the orbits, and a continued shortening of the limbs. In theory, each of these adaptations makes the modern bison a better grassland grazer (McDonald 1981).

Understanding the succession of various bison species and subspecies through the Holocene has proven difficult. Throughout the Mid-Holocene, *B. antiquus* and *B. occidentalis* seemingly shared an allopatric existence with the former ranging in the south and the latter in the north. The description of possible intermediate forms, and the occurrence of both forms together in at least one archaeological context, suggests a clinal gradation between the two (Frison 1991). Whether or not both forms contributed to the evolution of *B. bison* has been debated (Wilson 1978; McDonald 1981; Frison 1991). At least a few researchers have suggested that the Late Holocene forms probably originated from *B. occidentalis*, and gradually expanded south into the former range of *B. antiquus*, as climate ameliorated after the Altithermal (McDonald 1981). Others have suggested *B. occidentalis* as the possible progenitor of *B. athabascae*, the wood bison. Interestingly, wood bison are slightly larger and somewhat more browse oriented than plains bison (McDonald 1981). In this scheme, *B. antiquus* either returns to the southern Plains as *B. bison* after the Altithermal; or both *B. antiquus* and *B. occidentalis* contribute to the evolution of *B. bison*. In any event, the smaller *B. bison* is the only form present on the plains after about 5,000 BP (Wilson 1978).

Bamforth (1988) has suggested that the biological constraints of the bison digestive system likely acted in concert with large-scale environmental changes to produce a series of interrelated morphological and behavioral changes in Holocene bison. Specifically, he argued that bison at the outset of the Holocene were adapted to an essentially cooler, moister Pleistocene environment with relatively diversified, yet abundant forage. As the climate began a general warming trend throughout the Mid-Holocene, forage distributions changed and seasonal pressures, similar to those affecting modern bison, began to appear on a larger scale. In this scenario, larger bison of the Early Holocene required larger overall quantities of forage than later forms. As Bamforth (1988:43) describes:

> "Smaller animals have lower total forage requirements because they have less mass to provide nutrients for. They thus need to spend less time eating, allowing them to spend more time searching for higher quality food."

These bison were thus constrained in terms of the time that they could devote to meeting their greater foraging requirements. This resulted in selective pressure for a more mobile form as quality forage became more dispersed. The result was smaller bison that were less constrained biologically and thus, able to range farther for adequate sustenance (Bamforth 1988).

Bamforth's (1988) model provides a framework by which behavioural differences between temporally distinct bison populations may be inferred. If indeed the older forms were restricted in mobility relative to modern taxa, their diets should be more indicative of plant distributions on localized scales. Morphologically modern bison seem to appear consistently in the archaeological record by 4,000-5,000 BP (Dillehay 1974; Wilson 1978). It seems reasonable to hypothesize that older bison were more sedentary in general, while more recent forms likely exhibited migratory patterns similar to modern or historic herds. If changes in morphology between extant and extinct bison taxa may be linked to differences in foraging behaviour, they may also by extension indicate other behavioural differences in terms of predictability of aggregation and migration:

> "Changing forage conditions appear to account quite well for the observed changes in bison morphology during the late Pleistocene and early Holocene. Individual bison became smaller and smaller, but apparently lived in herds that became larger and larger, and more and more mobile over time...In addition, the steady decrease in the length of the period of maximum forage production within a year would have led to increasing seasonal differences in herd size...(and) declining forage production must have steadily reduced the numbers of animals in the region" (Bamforth 1988:149).

Evidence for prehistoric bison population demography is largely secondary; determined by the analysis of changing frequencies of individual animals from specific archaeological contexts, the relative frequencies of archaeological sites, and the archaeological evidence for a changing concentration of prehistoric peoples on bison as a resource. Mass communal drives appear as spontaneous or fortuitous events in the Paleoindian record. Examples that exist tend to be removed from each other in time and space (Forbis 1992). Early Mid-Holocene sites seem to hover on the fringes of the plains and occur in a notable paucity. Some have suggested that this apparent

"abandonment" of the Northern Plains during the Mid-Holocene, is simply an artifact of bias through sampling and poor preservation (Reeves 1973). In any event, "the Early Middle Prehistoric period on the Northern Plains appears to involve small-scale, single-component campsites" (Walker 1992:130). Most agree that during the Altithermal "there seems little doubt that the climate was not too favourable for the propagation of bison herds, except in possible oasis-like areas such as the Black Hills of Wyoming and South Dakota" (Frison 1991:191; see also Frison *et al.* 1976).

By 5,000 BP, however, it appears that favourable conditions returned, and it can be demonstrated through measurements that the bison present are the modern forms (Frison 1991). "With the close of the Altithermal, sites sprang up suddenly and everywhere on the northern plains, indicating a substantial increase in population and expansion onto the rolling grasslands." (Forbis 1992:48). Communal bison procurement reached its peak, at least in terms of numbers of animals killed, during the Late Prehistoric period (Frison 1991).

Walker (1992) compiled a database of radiocarbon dated cultural occupations from northern plains sites during the Mid-Holocene climatic optimum. These data clearly demonstrate a depression in the overall average of identified components between about 7,000 and 6,000 BP. It has long been assumed that at least one major factor contributing to this paucity was a decrease in bison population density on the Northern Plains resulting from poor forage conditions. In contrast, the Late Prehistoric is considered to be the age of communal bison hunting. In fact, the Late Middle Prehistoric may have seen the cultural climax of bison procurement with the "Besant" cultural complex (2,000 – 1,100 BP) (Dyck 1983; Forbis 1992). Besant managed to leave behind more numerous and widespread remains than any other single complex in Saskatchewan (Dyck 1983; Forbis 1992). It is interesting to note that another period of warm, dry climate known as the Scandic (1,700 – 1,100 BP) is thought to have occurred almost contemporaneously (Dyck 1983; Vickers 1986). Whether it was because of a favorable climate which fostered an increasing bison population, or because of technological and cultural developments, Besant cultures were highly succesful (Dyck 1983). By the end of the Late Prehistoric, bison populations in the Northern Plains are

thought to have approached the millions of individuals noted in Historic times (Roe 1951).

1.12 The Application of Combined Isotope Analyses to Paleoecology

Archaeologists attach great importance to understanding the prehistoric ecology of bison on the North American Great Plains. The dependence of prehistoric peoples on bison for a wide array of physical, social and spiritual needs necessitated a keen awareness of patterns associated with the movements and social behaviour of these animals (Bamforth 1988). A better understanding of bison ecology throughout the Holocene may allow for a better analysis of changes in social organization among prehistoric human groups (Frison 1991). In addition, such knowledge may have direct application to the management of modern bison herds, and the re-introduction of the species into their previous natural range (Kay and White 2001; White *et al.* 2001).

While modern analogy and historic observation offer the most detailed information readily available to the study of bison as a species, these sources are temporally removed from the vast expanse of bison prehistory (Larson *et al.* 2001). Prehistoric bison are primarily studied through detailed analysis of their skeletal remains, and via the examination of the distribution of these remains through time and space. New technologies, like the determination of stable isotope ratios in bone, allow for applied analyses of tissue composition, which may be used to infer diet (Cannon 2001). Targeting specific fractions of bone, such as collagen, may provide a time-integrated measure of dietary factors in long-lived herbivores like bison, over the better part of their lives (Chisholm 1989; Tieszen 1994). Finally, this dietary information may be used to infer aspects of bison ecology, such as behaviour and habitat climate (Chisholm *et al.* 1986; McKinnon 1986, 1990; Tieszen 1994; Jahren *et al.* 1998; Gadbury *et al.* 2000; Langemann 2000; Leyden and Oetelaar 2001; Larson *et al.* 2001; Brooks-Lovvorn *et al.* 2001).

Such inferences have proven problematic, however. Most isotopic studies of bison bone have focused upon the analysis of carbon isotope ratios as they relate to the botanical composition of diet. While most researchers agree that carbon isotope values in herbivore tissue reflect dietary composition, there is great debate as to how these data

may be interpreted. Specifically, does the composition of bison diet directly reflect climatically-related distributions of forage, or does the foraging behaviour of the species complicate this relationship? The key to this problem has been the degree to which bison behaviour may be consistent or at least predictable (Bamforth 1988). Modern and historic studies of bison ecology have often proven contradictory. However, examination of these sources seems to reveal that an understanding of bison behaviour may be limited to the generalization that bison seem to make adaptive choices designed to minimize the negative impacts of their environmental circumstances and maximize the beneficial ones. While bison foraging patterns are tied to forage distributions in the context of local and seasonal climate, their specific foraging behaviour will vary depending upon unpredictable factors like fire, and limiting factors such as population demography, regional carrying capacity, and local topography (Larson 1940; Vinton *et al.* 1993; Griebel *et al.*1998; Truett *et al.* 2001; Hart 2001).

Another important consideration is that recent bison forms appear to be well adapted to the North American grasslands, in particular, the short-grass plains (McDonald 1981). Within this context, they have been noted to withstand a wide variety of changing forage conditions and quality. Their own forage preferences and tendencies seem to harmonize well with the seasonal cycles within this ecoregion (McDonald 1981; Bamforth 1988). It is often only when the environment makes it difficult to obtain adequate sustenance that they are found in sheltered areas or at the region's peripheries (Moodie and Ray 1976; Morgan 1980; Epp 1988). Thus, most of the behavioural variation between bison groups, as seen within the recent and historic past, is ultimately attributable to habitat variation on seasonal and regional scales.

It seems reasonable to accept a model in which modern Canadian plains bison move seasonally, between the parklands and plains, but less so in moderate winters (Malainey and Sherriff 1996). Nevertheless, bison bone isotope compositions should not record short-term deviations as a result of seasonal movements, due to the slow turnover of bone collagen (Tieszen 1994). Because of the integrated compositions of their tissues and because bison cannot generally be selective when grazing, the isotopic values isolated from bison bone tissue should represent an average of the dietary sources within their foraging range (Tieszen 1994). Only significant deviations over an

extended period should have a measurable impact on the isotopic composition of bison bone tissue. Differences in reconstructed diet through time may thus be explained by either significant changes in either climate or foraging behaviour or both.

Studies of prehistoric bison populations must account for one more consideration. While modern and recent bison are creatures of the short-grass plains and exhibit a continuum of behavioural responses within this context, bison of the Early and Mid-Holocene may exhibit a fundamentally different ecology. This discrepancy may encompass significant differences in both physiology and behaviour. Bison of the Pleistocene, from which more recent forms are derived, exhibited several different morphologies and were likely adapted to different environmental niches (McDonald 1981; Guthrie 1966, 1970; Wilson 1978; Bamforth 1988). It has been assumed that as the area of the current Great Plains underwent a transition from a wooded savanna to the expanded modern grasslands, many of these forms had difficulty surviving (McDonald 1981; Forbis 1992).

A combination of environmental change and possible human over-exploitation resulted in vastly reduced numbers and the survival of only one bison species into the Holocene. Throughout the last 10,000 years, a gradual but continuous "dwarfing" of bison forms has occurred, and although the Holocene has been climatically stable in comparison to the Pleistocene, this "dwarfing" trend may have slightly accelerated during the period of Mid-Holocene warming known as the "Altithermal" (Wilson 1978). In addition, bison populations which were slowly growing after the Pleistocene/Holocene transition may have stagnated or even decreased during this period (Dillehay 1974; Forbis 1992).

Archaeological evidence and comparative herbivore anatomy have led some to speculate that these larger, Early Holocene forms may have been more sedentary and less gregarious than modern Plains bison (McDonald 1981; Bamforth 1988). In addition, they may have exhibited a higher degree of browse-oriented foraging behaviour (McDonald 1981; Frison 1991; Forbis 1992). Slow population growth during the first half of the epoch and a comparative explosion in the last 3,000 years, as demonstrated by the massive numbers of animals during historic times, may also

indicate adaptive differences between recent and ancient forms. As these points illustrate however, these changes may also be predictable in a general sense.

Carbon isotope analyses of dietary forage inputs from bison bone would benefit from restricting either the temporal or spatial scope of studies. Temporal comparisons of bison diet should try to restrict themselves to as tight a regional focus as possible. Similarly, spatial studies should concentrate on tightly controlled temporal periods. Unfortunately, these are "best case" scenarios and do not often reflect the reality of archaeological and paleontological data.

When comparing stable isotopic data to a general ecological model of bison behaviour within either of these frameworks, it is highly desirable to control for other complicating factors. While climate variations often motivate specific responses from bison through an impact on forage distributions, the nature of this relationship may have changed through time (Bamforth 1988). By controlling for a changing climate, even in a general sense, an ecological model may begin to make simple inferences about variations in behavioural response to specific climate conditions. Unfortunately, most records and archives of Holocene climate in North America are either very general (large scale trends) or very specific. It is often problematic to associate archaeological materials directly with sources of proxy paleoclimatic data. Isotopic data from archaeological bone collagen represent extremely specific periods in time no greater than the lifespan of the animals being studied (Chisholm 1989). Even when proxy data can be obtained from the same archaeological context as that of the sample bone, they may only yield a very general interpretation of past climate.

Analysis of hydrogen or oxygen stable isotope ratios from prehistoric bison bone may provide a contextually derived climate record (Cormie 1991; Cormie *et al.* 1994a, 1994c). The hydrogen and oxygen isotope composition of environmental waters from within the continental interior are largely a function of temperature and relative humidity in the local region (Gat 1996). The isotopic compositions of herbivore tissues are primarily derived from local dietary and drinking water sources (Cormie *et al.* 1994c). Thus, the hydrogen and oxygen isotope composition of bison tissues should directly relate to the local climate (Cormie *et al.* 1994a, 1994c). Hydrogen isotope compositions may be particularly useful as the stable isotopes of hydrogen exhibit the

largest mass differences of all biologically important elemental isotopes (Hoefs 1973). As a result, the hydrogen isotope composition of waters within different climatic contexts may be highly differentiated and thus, the isotopic composition of animal tissues from different ecological contexts should be significantly different.

The analysis multiple elemental isotope systems allow the researcher to examine disparate portions of an animal's ecological context which are directly related in time and space. A researcher may then compare hypotheses generated by predictive models of animal behaviour to the information directly derived from the tissues of animals from different time periods or spatial regions. Such comparisons may initially be qualitative rather than quantitative. They may only indicate the direction of change, or relative magnitudes of change, but they may nonetheless, allow for examination of the relationships between variables. For example, a key difficulty in carbon isotopic analyses of bison diet has been uncertainty related to whether or not dietary changes over time or between areas were primarily related to changes in environmental factors altering forage distributions, or some difference in foraging behaviour (Tieszen 1994). By controlling for climate change using contextually derived climate data (i.e. hydrogen isotope ratios), it may be possible to predict whether or not changes in forage distribution would be expected. Dietary carbon and nitrogen values could thus be interpreted on the basis of whether or not forage distributions have significantly changed. In cases where climate may not adequately explain observed change or stability in diet, it may be possible, but simplistic, to compare temporal and spatial theories of bison behaviour to the isotopic data.

The ability to make these interpretations is heavily reliant upon a concrete understanding of the controlled variables. Thus, it is necessary to demonstrate the solid and direct relationship between the proxy source of context, and the ecological variable which one seeks to control. For example, the relationship between the hydrogen isotopic composition of consumer tissues and climate must be established before such data may be used to approximate climatic context (Tieszen 1994).

The degree to which other elemental isotope systems may contribute to the understanding of past ecologies is unknown. Nitrogen isotope ratios may vary within species through time and over distances from the influence of a variety of biological or

ecological phenomena (Ambrose 1991). While most of these inter-specific determinants have a link to climate in the overall habitat, the specific mechanism by which differences in the isotopic composition of animal tissues occur has remained elusive. We may, nevertheless, be able to make general comparisons between the nitrogen isotopic composition of tissues and the isotopic composition of other isotope systems within these tissues, as they relate to specific ecological contexts. For example, does change or stability in the nitrogen isotope composition of the tissues of different animals correlate with changes in diet or environment as evidenced by carbon and hydrogen values? If not, are there other biological or ecological conditions or trends, which may theoretically affect tissue composition? How do these theories compare to models of an animal's behaviour and/or biology through time and space?

1.13 Objectives and Thesis Structure

The goal of this research is to add to available knowledge concerning the prehistoric ecology of bison through time, and suggest a new approach to this goal using a combination of stable isotope data. Within such a framework, the first objective is to determine whether or not δD values of bison bone collagen can be used to illustrate environmental differences between archaeological sample groups, from different times, within a restricted regional context. If proven practical, such data would allow for the generation of general climate models against which other isotopic data derived from the archaeological samples could be gauged. The second objective is to develop inferences into prehistoric bison ecology through time, by comparing and contrasting the stable hydrogen, nitrogen and carbon isotope compositions of these samples. Such interpretations will be assisted by comparison to current theoretical models of Northern Plains Holocene climate change and prehistoric bison behavioural ecology. Finally, these isotope-generated interpretations of prehistoric bison ecology will be used to discuss the adaptation of prehistoric human populations to changing bison populations and climate.

Chapter 1 presents background data relevant to the thesis. This includes: a discussion of bison remains in archaeological studies, the carbon, nitrogen and hydrogen isotopic analysis of bone collagen, considerations for the study of bison

ecology through time and space, and the theoretical basis of the thesis. The next three chapters are written as individual papers examining different aspects of the thesis objectives. Chapter 2 details the results of stable hydrogen isotope analysis of modern and prehistoric bison bone. Chapter 3 examines how determining the carbon, nitrogen and hydrogen isotope composition of prehistoric bison bone may be used cumulatively to examine the prehistoric ecology of bison through time in southern Saskatchewan. Chapter 4 examines how a better understanding of regional bison ecology through time, based on isotopic analyses of prehistoric bison bone, may be applied to the study of prehistoric human groups in Saskatchewan and the northern Great Plains. In the final chapter, I summarize my findings, address any problems or limitations of my approach, and suggest directions for future research. It should be noted that both the Amisk and Thundercloud site materials were excluded from this segment of the analysis due to their low samples sizes.

A significant amount of relevant background data is detailed in several appendices to the body of this thesis. Appendix A details the archaeological and modern sites from which the samples used for analysis were collected. Appendix B presents the results of a radiocarbon dating assay involving a single sample from each archaeological context. Appendix C contains relevant information concerning each sample used in this study, and related data catalogued for each specimen. Appendix D provides a detailed summary of the techniques and methods used to analyze each sample including; cleaning, collagen extraction, combustion, cryogenic separation and gas collection, and mass spectrometry. Appendix E provides data on the integrity of each sample and a discussion of the criteria used for selection. Appendix F presents the reported stable isotope ratios for all samples examined in this project. Appendix G details the procedure for calculating the proportion of C_4 grass in the diets of the various sampled bison from the measured $\delta^{13}C$ value of their bone collagen.

Chapter 2. CLIMATE INFERENCES FROM THE STABLE HYDROGEN ISOTOPE ANALYSIS OF ARCHAEOLOGICAL BISON BONE

2.1 Introduction

Across the North American Great Plains, the skeletal remains of prehistoric bison are frequently recovered from archaeological sites. Because these bones are often contemporaneous with an associated prehistoric human population, they have value to the study of both human and animal paleoecology. Stable carbon isotope analysis of bison bone collagen has been previously used to infer bison diet and by extension, paleoclimate (McKinnon 1986, 1992; Leyden and Oetelaar 2001; Brooks-Lovvorn *et al.* 2001). However, questions have been raised as to whether dietary inferences based on carbon isotope data are representative of actual paleoclimate parameters, or whether these indicators are complicated by the movements and foraging behaviour of the animals themselves. The comparative analysis of stable hydrogen isotope ratios from bison bone collagen may represent a new method by which a more direct examination of relative temperature and climate change through time is possible.

In arid, continental ecosystems, temperature is the primary determinant of the hydrogen isotope composition of meteoric and biologic waters (Dongmann *et al.* 1974; Gat 1996). As air masses dry and move into the continental interior, they become the dominant source of local moisture. In the absence of large bodies of surface water or significant humidity, temperature at the site of precipitation as correlated with season, latitude and elevation primarily determines the δD composition of rain (Ehleringer and Dawson 1992; Schoetterer *et al.* 1996). Temperature changes also control the rate of evaporation in soil and surface waters, as well as evaporative transpiration from the leaves of plants that derive water from these soils (Gat 1996). Higher temperatures act to increase evaporation and evapotranspiration rates and subsequently, cause enrichment in the deuterium composition of water in these substrates. The tissues of

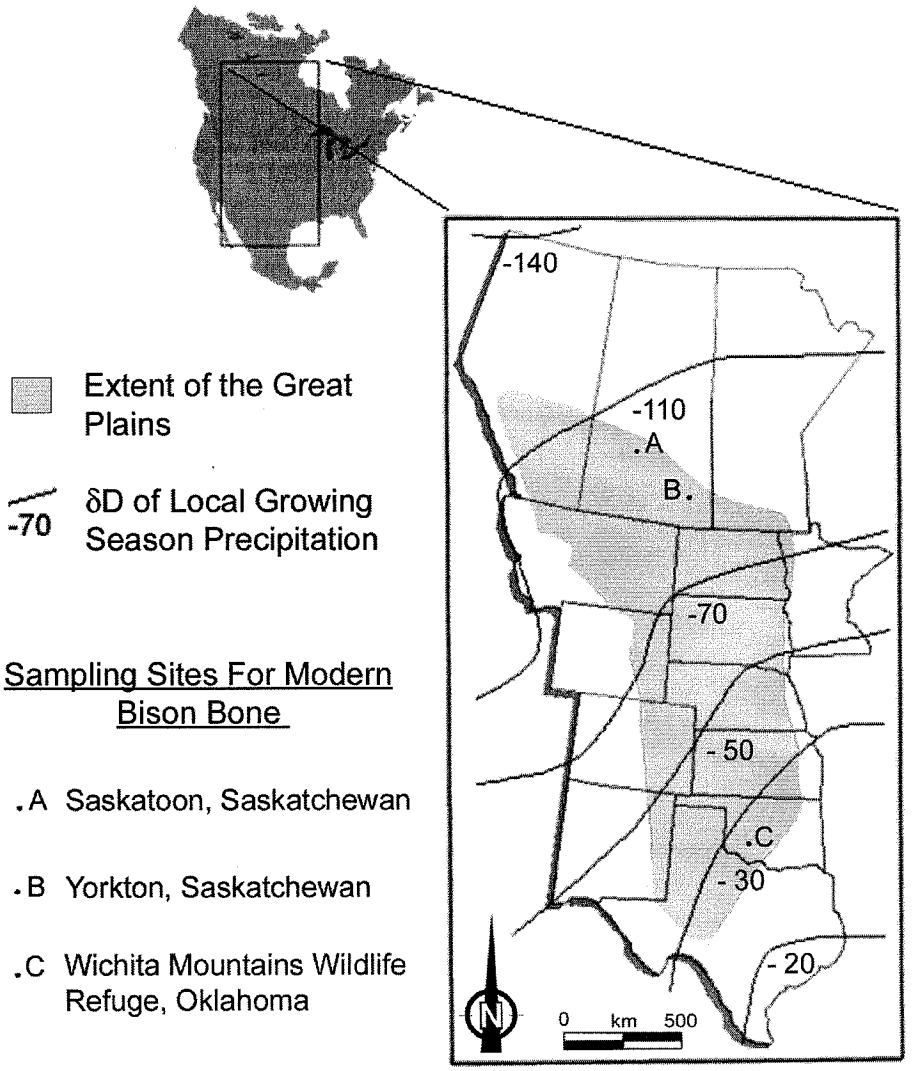
consumers that obtain dietary and drinking water from these sources should reflect their isotopic compositions (Cormie *et al.* 1994c). As a result, they ultimately record the cumulative impact of local temperature on several ecological reservoirs. In addition, tissues like bone collagen, that are biochemically inert following synthesis in living organisms, provide an aggregate record of an animal's diet over most of its life. Thus, short-term variations in isotopic composition, such as seasonal differences, are averaged.

Growing-season precipitation forms gradients of decreasing δD composition from southeast to northwest across the Great Plains (Hobson and Wassenaar 1997) and bone collagen from geographically distinct groups of modern bison reflects this (Figure 2.1, see Table 2.1). Although bison bone collagen is enriched in deuterium relative to growing season rain, a spacing of approximately 70‰ observed between the δD values of collagen from southern Saskatchewan bison and Oklahoma bison seems to be consistent with the isotopic composition of growing-season precipitation for both regions. Cormie *et al.* (1994a, 1994c) describe a similar relationship between the δD composition of growing-season rain and modern deer bone collagen across North America.

Despite the potential for paleoclimate reconstruction (Miller 1984; Cormie *et al.* 1994a, 1994c), researchers have yet to attempt such inferences from fossil animal remains. There is a debate concerning the potential causes of δD variation within ecosystems that are not directly controlled by climate. Physiological differences between certain plant species can cause variation in the δD composition of their tissues (Sternberg 1989). There is also an uncertainty concerning the relative roles of diet versus drinking water in the overall composition of animal tissues (Hobson *et al.* 1999). Until the relationship between the isotopic composition of diet and drinking water with that of animal body tissues is better understood, quantitative reconstruction of paleoclimatic parameters will not be possible.

Fortunately, the analysis of tissues such as bone collagen, which provide aggregate estimates of dietary and drinking water sources over the lifetime of long-lived mammals like bison, should be comparable through time if an effort is made to restrict

Figure 2.1 Average Patterns of δD Composition in Growing-Season Precipitation From Across The Great Plains (after Hobson and Wassenaar 1997)

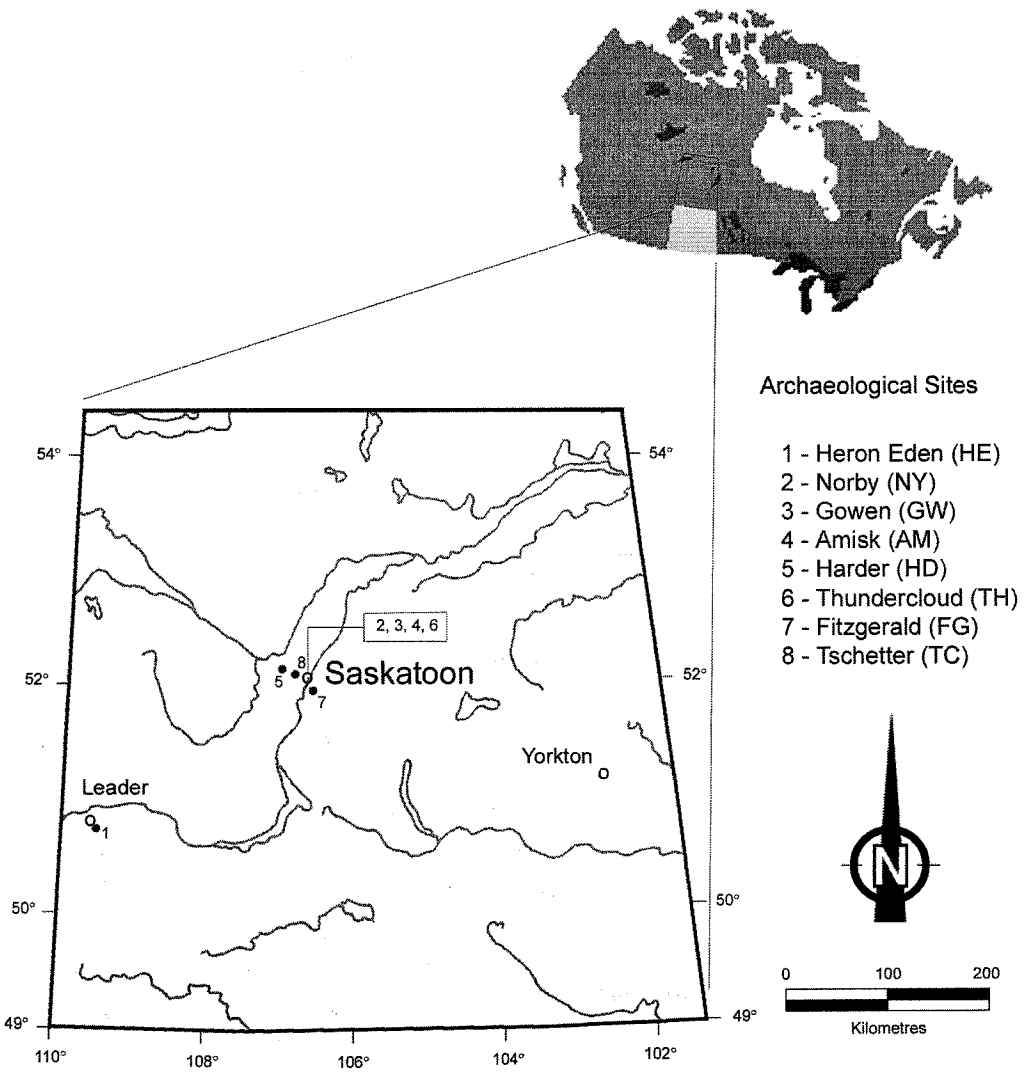


the spatial extent of sampling (Cormie 1991). Furthermore, bison do not typically consume those species of plants which are highly differentiated in terms of hydrogen isotope composition (Peden 1976; Sternberg 1989). As a result, changes through time in the proportions of specific forage species that they consume will not affect the δD composition of their tissues. Therefore, it should be possible to infer relative differences in past environmental temperatures from the tissues of different groups of fossil bison. The goal of this study is to examine climate change through time as indicated by variations in the δD composition of temporally distinct groups of bison from Southern Saskatchewan and subsequently evaluate these inferences by comparing them to other proxy paleoclimate data for the region.

2.2 Materials and Methods

Samples of bison bone representing seven modern individuals and 52 prehistoric animals were chosen for this study. Selection of individual bison for analysis, representing the minimum number of individual (MNI) animals for each prehistoric cultural component, was accomplished by only sampling replicate elements from the same side of each animal. Modern bison bones were obtained from two Great Plains locations that exhibited distinct precipitation δD values. Four modern bison from southern Saskatchewan (Saskatoon and Yorkton) and three bison from Oklahoma (Wichita Mountains Wildlife Refuge) were sampled. All modern bison were raised on forage and water obtained locally. Prehistoric samples were collected from eight different archaeological sites previously excavated in southern Saskatchewan. These sites range in age from approximately 10,300 to 1,050 years BP and with the exception of the oldest, all occur within a 50 km radius around the city of Saskatoon (Figure 2.2). Following collagen extraction, combustion and gas separation, eight samples of CO_2 gas representing each of the prehistoric cultural components were sent to the Rafter Radiocarbon Laboratory in New Zealand for AMS dating. Usable dates were obtained for seven of the sites and those dates are used and reported here (see Table 2.1). The sample from the Tschetter site was contaminated and thus, an average of earlier radiometric dates is used instead (Linnamae 1988).

Figure 2.2 Map of Southern Saskatchewan Showing the Location of Archaeological Sites Used For This Study



Bone samples of about 3g were selected from each specimen for stable isotope analysis. Each sample was inspected, mechanically cleaned, and washed in a hydrosonic bath to remove dirt and other particulate matter. Bone collagen was then extracted and purified according to the method of Sealy (1986). All modern samples were first treated with a 2:1 methanol/chloroform solution to remove lipids. All samples were then de-mineralized in 0.25 M HCl at room temperature. Each was then rinsed to a neutral pH using de-ionized water and treated with 0.125 M NaOH at room temperature for 7 hours if modern, and 20 hours if archaeological, to remove humic and organic acids. Next, all samples were again rinsed to neutral pH using de-ionized water, frozen and freeze-dried. Prior to combustion, each was ground and homogenized using an analytical mill.

A portion of the total hydrogen in bone collagen is available for isotopic exchange with ambient water vapour H (DeNiro and Epstein 1981a; Wassenaar and Hobson 2000). As a result, it was necessary to quantify and eliminate the effect of this uncontrolled, temperature dependant variable. The proportion of exchangeable hydrogen was first determined using a static equilibration technique with steam having a wide range of hydrogen isotopic values (-135 ‰, +115 ‰, +525 ‰) at a constant temperature (135°C) for two hours followed by measurement of the resulting δD values (see Wassenaar and Hobson 2000). Samples of modern Saskatchewan bison bone collagen were used for this quantification. The hydrogen available for isotopic exchange at this temperature was determined to be 20.0 ± 2.5% (n = 4). This proportion is consistent with other published estimates for various biological tissues (Schoeller *et al.* 1986; Cormie *et al.* 1994b; Hobson *et al.* 1999). Subsequently, samples were standardized by equilibration with steam of known δD value (δD = -135‰) at 135°C for two hours and calibrated according to the determined portion of exchangeable hydrogen (see Wassenaar and Hobson 2000).

After equilibration in Vycor breakseal tubes, all water vapour was removed cryogenically. Samples were then sealed under vacuum and combusted at 850°C in the presence of cupric oxide, followed by cryogenic separation of CO_2 and N_2 from H_2O. Waters of combustion were reduced to H_2 gas by using hot zinc, and D/H ratios were measured on a Micromass Optima dual inlet isotope-ratio mass spectrometer. Stable

hydrogen isotope results are reported in parts per thousand (‰) deviation from the Vienna Standard Mean Ocean Water Standard (VSMOW).

2.3 Results

The stable hydrogen isotope data from this study demonstrate that significant differences exist between the hydrogen isotope composition of bison bone from groups of animals foraging in the Saskatoon area at different times during the Holocene (Table 2.1; ANOVA, $F = 5.1794$, $df = 7,44$, $P < 0.001$). The mean of δD values from each time period are distributed across an overall range of approximately 19‰. A multiple comparison analysis (Tukey's HSD test) was used to identify where significant differences occurred. Bone from the Fitzgerald site population had the most positive δD (-115 ±8.0‰) and differed significantly from the Gowen ((-132 ±7.3‰) $P < 0.01$), Harder ((-132 ±10.6‰) $P < 0.01$) and Thundercloud ((-134 ±10.8‰) $P < 0.05$) site populations. Bison from the Norby site also had significantly higher δD values (-117 ±7.7‰) than both the Gowen ($P < 0.05$) and Harder sites ($P < 0.005$). Mean δD values for bison from some of the prehistoric sites (Gowen, Harder and Thundercloud) were close to values expected for modern bison (-131 ±9.4‰) that live in southern Saskatchewan. Others (Heron Eden, Amisk, Tschetter) gave intermediate values.

Significant differences were also evident when modern Saskatchewan bison were compared with prehistoric bison (ANOVA, $F = 5.35$, $df = 8,47$, $P < 0.0001$) and when all Saskatchewan groups, modern and prehistoric, were evaluated against modern specimens from Oklahoma (ANOVA, $F = 21.2$, $df = 9,49$, $P < 0.0001$). The δD composition of bison bone from the three most positive groups were found to be significantly different from expected modern values (Norby $P < 0.01$, Fitzgerald, $P < 0.01$ and Tschetter, $P < 0.05$; Dunnett's test). Additionally, all modern and prehistoric bison samples from Saskatchewan were significantly enriched in deuterium relative to modern specimens from Oklahoma ($P < 0.001$; Dunnet's test).

Table 2.1 Mean δD Values of Bison Bone Collagen From Modern Locations and Prehistoric Archaeological Sites

Site	n	MNI	Cultural Association *	Species	^{14}C yr. BP	yr. BP	Lab #	Mean δD
Prehistoric								
HE	7	7	Cody	*B. antiquus*	9,168 ± 50	10,352 ± 134	NZA 15745	-127 ± 4.0
NY	7	7	Mummy Cave	*B. occidentalis*	7,036 ± 45	7,846 ± 103	NZA 15747	-117 ± 7.7
GW	6	6	Mummy Cave	*Bison sp.*	5,863 ± 55	6,653 ± 143	NZA 15746	-132 ± 7.3
AM	2	2	Oxbow (Level 4)	*B. bison*	4,358 ± 45	4,941 ± 103	NZA 15748	-123 ± 3.5
HD	8	8	Oxbow	*B. bison*	4,221 ± 45	4,823 ± 33	NZA 15776	-132 ± 10.6
TH	3	3	McKean (Level 5)	*B. bison*	3,382 ± 55	3,804 ± 13	NZA 15749	-134 ± 10.8
FG	10	10	Besant	*B. bison*	1,563 ± 45	1,442 ± 101	NZA 15750	-115 ± 8.0
TC	9	9	Old Woman's	*B. bison*	1,035 ± 40 **			-121 ± 8.8
Modern								
Saskatchewan	4			*B. bison*	Modern			-131 ± 9.4
Oklahoma	3			*B. bison*	Modern			-67 ± 11.4

^{14}C yr. BP, "conventional radiocarbon years before present"
Yr. BP, "calibrated radiocarbon years before present", 2 sigma range, calibrated using INTCAL98_14C, based on Stuiver *et al.* 1998
Lab #, Rafter Radiocarbon Laboratory sample number
* Cultural associations are based upon projectile point associations (see Corbeil 1995; Zurburg 1991; Walker 1992; Amundson 1986; Dyck 1977; Webster 1999; Hjermstad 1996; Linnamae 1988). For the multi-component sites (Amisk and Thundercloud) the corresponding excavation level from which the sampled bones derive is indicated
** The Tschetter date is an average of radiometric radiocarbon dates determined prior to this study (see Linnamae 1988)

2.4 Discussion

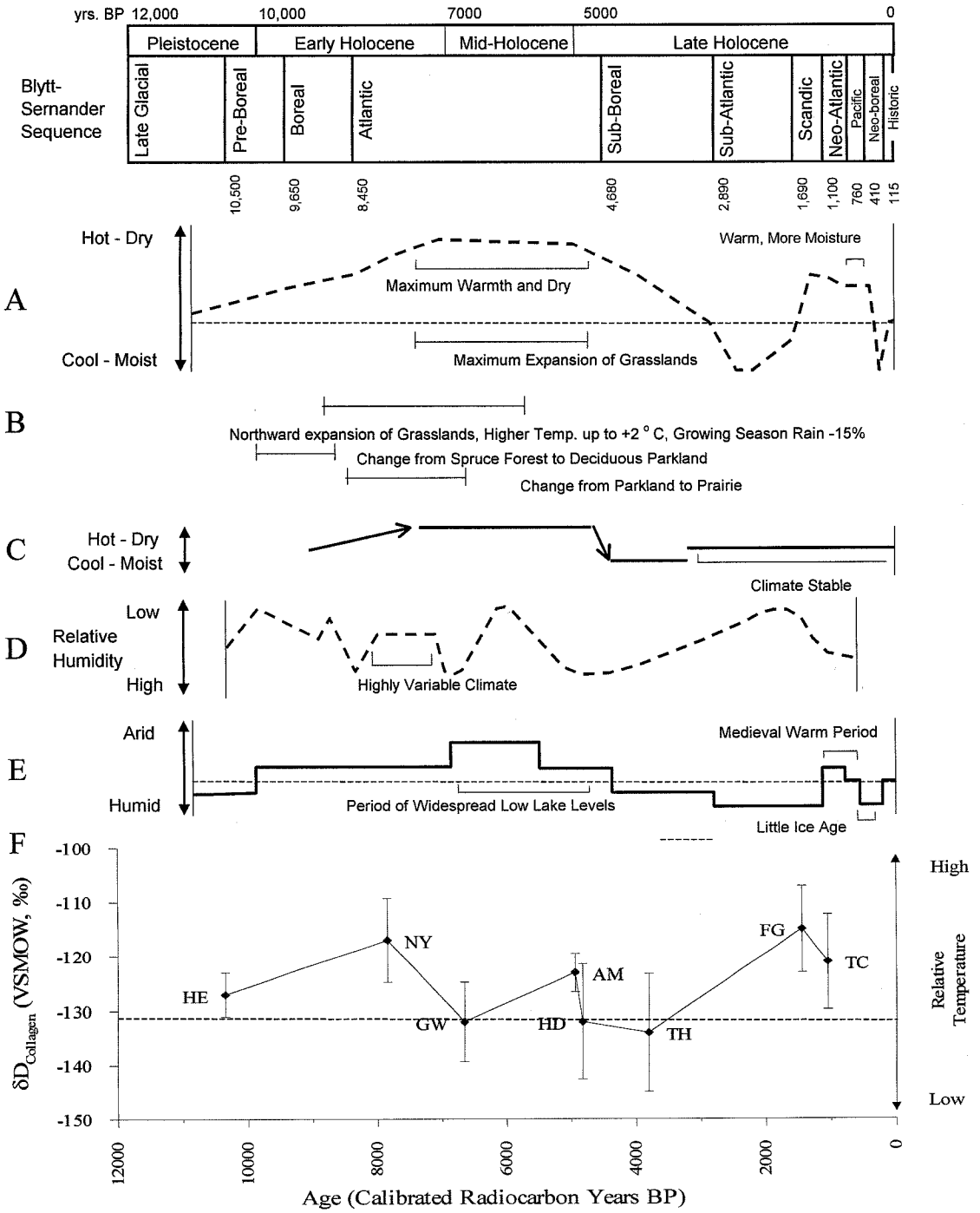
2.4.1 Holocene Climate Change

Several independent and associated lines of evidence, including studies of palynology (Sauchyn and Sauchyn 1991; Vance *et al.* 1995), mineralogy (Shang and Last 1999), macrofossils (Yansa and Basinger 1999), diatoms (Wilson and Smol 1999), stable isotopes and algal pigments (Van Stempvoort *et al.* 1993) indicate the occurrence of dramatic climate changes throughout the Holocene in the northern Great Plains and Saskatchewan (Figure 2.3). The first half of the Holocene is largely considered to have been warmer and drier than the last half; although certain episodes during the last 5,000 years probably reached temperatures and levels of aridity that were comparable to those earlier in the epoch (Dyck 1983; Lemmen and Vance 1999). It should be noted that verifying the occurrence specific climate events through analysis of multiple proxies from several locations is difficult. Varied levels of temporal and spatial resolution pose limits on chronological control (Lemmen and Vance 1999). In addition, Holocene climate change seems to transgress time, occurring progressively later from southwest to northeast across North America and the Great Plains (Walker 1992; Vance *et al.* 1995; Sauchyn 1997). For these reasons, it has been recommended that comparisons within regions be made to the closest available sources of proxy data (Beaudoin 1993). Even at these smaller scales a wide range of climate conditions may occur and thus, only general interpretations of climate may be possible.

During the Late Pleistocene and Early Holocene (~10,500 to 7,000 years BP) the Northern Great Plains experienced a period of warming climate pursuant to the conditions at the end of glaciation. In Saskatchewan, although this period is poorly understood relative to later ones (Dyck 1983), evidence of periodic aridity has been described by several researchers (Lemmen and Vance 1999). The transitional nature of climate during this time period is supported by pollen (Sauchyn 1997) and macrofossil (Yansa and Basinger 1999) data that suggest a gradual change in the vegetative composition of the southern part of the province from a spruce forest to deciduous parkland.

Higher temperatures and reduced aridity characterized the Atlantic climate episode (~8,500 to 4,600 years BP). The Middle Holocene (~7,000 to 5,000 BP)

Figure 2.3 Holocene Climate Change in Southern Saskatchewan: A Multi-Proxy Comparison With δD Ratios in Bone Collagen From Bison in Different Time Periods



Error Bars Represent One Standard Deviation ------- Modern Conditions

A - adapted from Dyck 1983; B - adapted from Vance et al. 1995, Vreeken 1999, Yansa and Basinger 1999

C - adapted from Sauchyn and Sauchyn 1991; D - adapted from Shang and Last 1999

E - adapted from Lemmen and Vance 1999; F - δD Results of This Study (2003)

occurred within this period and is considered to be concurrent with the time frame in which Atlantic conditions reached a maximum. This is often referred to as the "Mid-Holocene climatic optimum" or "Altithermal". Widespread low lake levels in Southern Saskatchewan and throughout the Canadian Plains during this time are recorded in the sedimentary records of several lakes suggesting increased temperature and/or aridity (Sauchyn and Sauchyn 1991; Sauchyn 1997; Lemmen and Vance 1999; Birks and Remenda 1999; Richmond and Goldsborough 1999). In addition, Yansa and Basinger (1999) use changing frequencies of recoverable plant macrofossils and evidence of increased fire frequency from the kettle-fill remnant of a Mid-Holocene pond near Moose Jaw, Saskatchewan to suggest a period of increasing aridity between 8,800 to 5,500 years BP. It is also during the Atlantic episode that grasslands are thought to have replaced the parklands in much of the Canadian Plains and subsequently expanded to reach their maximum extents to the north and east (Vance *et al.* 1995; Vreeken 1999).

The Late Holocene (5,000 years BP to present) is thought to have begun with an amelioration of the climate, eventually making a transition into cooler and wetter conditions than present between about 3,000 and 2,000 years BP (Dyck 1983; Vance *et al.* 1995; Lemmen and Vance 1999). Subsequent periods of warmth and aridity like the "Medieval Warm period" (1,100 to 800 years BP) and cold, humid episodes such as the "Little Ice Age" (500 to 100 years BP) are also recorded by several sources (Sauchyn and Beaudoin 1998; Lemmen and Vance 1999).

Pollen cores and sediment records from lakes in the Cypress Hills region of the southwest portion of Saskatchewan give little indication of vegetative and hydrological changes associated with either the Medieval Warm Period or Little Ice Age (Sauchyn and Sauchyn 1991; Wilson and Smol 1999). Today, this area is largely forested and considered to be ecologically distinct from the grasslands that typically characterize the southern part of the province (Acton et al. 1998). More northerly locations do record these climate changes. At Redberry lake, 80km north of Saskatoon, Van Stempvoort *et al.* (1993) attempted to interpret the sedimentary record for the last 2,400 years. Using data derived from mineral studies, stable isotope analyses, and the interpretation of pigments, they proposed the presence of a period of warm/dry conditions from about

1,100 to 900 BP which they correlate to the Medieval Warm Period. They also suggest the occurrence of a warm, dry climate between about 2,500 and 1,500 BP.

2.4.2 Hydrogen Isotope Data

Differences in Holocene climate as reconstructed from the tissues of prehistoric Saskatchewan bison are roughly comparable to regional schemes of climate change in Saskatchewan as inferred by other proxy data. Bison bone collagen from both the Norby and Fitzgerald sites display δD values that are significantly more positive than the observed values for modern bison in southern Saskatchewan. They are also distinct from at least three of the other prehistoric sites. This suggests that the dietary and drinking water sources utilized by the Norby and Fitzgerald bison were enriched with deuterium relative to those used in other time periods. Because higher D/H ratios in meteoric waters primarily correlate with increased temperature at the site of precipitation, it may be assumed that bison from the Norby and Fitzgerald sites obtained diet and drinking water in a warmer climate.

At 7,800 years BP, the warm and arid conditions associated with the Atlantic Climate Episode dominated southern Saskatchewan and had an impact on the Norby site bison. This interval of maximum temperature and minimum moisture is well documented in both Saskatchewan (Dyck 1983; Lemmen and Vance 1999), and the Northern Great Plains (Vickers 1986; Walker 1992; Bamforth 1989). Similarly, warmer temperatures associated with the last two millennia of the Late Holocene, likely affected bison from the Fitzgerald and Tschetter sites. Records of paleoclimate from across Southern Saskatchewan are variable for this time period. Outside of the Cypress Hills, however, warm and/or dry conditions associated with the Medieval Warm Period are consistently reported (1,100 to 800 years BP). In addition, at least one record of extended heat and/or aridity dating to around 2,500 – 1,500 years BP, has been reported at Redberry Lake, near Saskatoon. Bison groups from both the Fitzgerald (1,450 BP) and Tschetter (1,050 BP) sites have mean δD values that are significantly more positive than modern bison.

With the exception of the Gowen bison, samples from the remaining sites (Heron Eden, Amisk, Harder, Thundercloud) may be considered to be comparable with

periods of moderate climate or those of environmental transition. The time periods represented by all of the sites examined in this study do not represent episodes in which regional climate is considered to have been significantly cooler and/or moister than modern conditions; and notably, all have slightly to significantly more positive δD means. Both the Harder site (4,800 BP) and Thundercloud site (3,800 BP) bison exhibit mean δD values that are close to the modern average suggesting that temperatures at this time were similar to present. This is plausible given the transitional nature ascribed to the Sub-Boreal episode (Dyck 1983). The high δD variability observed within the Thundercloud sample group is conspicuous. The low sample size (n=3) of the group may be to blame. In addition, a compression of contextual stratigraphy was noted during excavation of the Thundercloud site and it is possible that some of the McKean bone samples represent more than one cultural occupation (Webster 1999).

Both the Harder and Amisk Oxbow cultural assemblages date close to the transition between the Atlantic and Sub-Boreal climate episodes. It is not surprising that the mean δD values for the Amisk bison are intermediate to modern values and to those of Norby. What is notable is the nearly 10‰ difference between the Amisk and Harder groups. This disparity suggests a large degree of climate change within a 100 to 200 year time interval. This transition is made all the more interesting by the fact that both sites have been ascribed to a common cultural tradition. Unfortunately, the limited sample size of the Amisk group (n=2) calls into question the relevance of this result and thus, any subsequent interpretations.

The Heron Eden site (10,350 BP) represents the earliest bison group sampled in this study. Although climate at the Pleistocene/Holocene transition is poorly understood, it is thought to be generally characterized by a gradual warming which continues into the Middle Holocene. The mean δD value of bone collagen from the Heron Eden bison is intermediate to that of modern bison and to those that here represent high temperature periods (Norby and Fitzgerald). This would be consistent with such an interpretation. However, the Heron Eden site is roughly 200km southwest of the area in which the other sites occur. As such, it is difficult to compare this result to the other sample groups. Although the temperature at this location may have been warmer 10,300 years ago, this does not necessarily mean that temperature was

equivalent in the Saskatoon area at the same time. Similarly, it can not be assumed that the relationships between climate variables which currently characterize these regions are the same.

Finally, cooler conditions at about 6,650 BP, as represented by the δD composition of bone collagen from the Gowen bison, are unexpected. Both Norby and Gowen date to within the Atlantic Climate Episode, which is generally considered to have been warm relative to today. Gowen occurs within the period usually associated with maximum temperature and aridity. Plant macrofossil evidence collected during the excavation of the Gowen sites suggests the association of adverse environmental conditions (Walker 1992). Although plant foods presumably grew sparse during the height of Mid-Holocene aridity, certain species may have thrived. Chenopod seeds in the Gowen site assemblage may indicate the presence of conditions similar to those which motivated an increase of Chenopodium and Amaranth during the droughts of the 1930s (Forbis 1992). Additionally, the collected assemblage of bison remains was highly fragmented and processed (Walker 1992). Whether having resulted from a lack of animal resources, or in prelude to the production of pemmican, a reliable, storable, portable and nutritious food source, this may have been an indication of some kind of stress that necessitated a change in resource utilization (Forbis 1992).

There are some possibilities that may aid in reconciling these data with the results of this study. Although, the Middle Holocene was generally warm, there is evidence to suggest periods of variability (Shang and Last 1999). Ultimately this interval was "episodic in nature and regionally variable in severity" (Walker 1992). Although the δD of bison bone collagen represents lifetime averages of regional dietary and drinking water inputs, the life-spans of bison are relatively brief in terms of large-scale climate processes. Shifts in climate significant and prolonged enough to differentiate the δD composition of the tissues from two groups of bison which are separated by very little time, are possible. The observed disparity between the Amisk site and Harder site bison may be representative of this. As well, it is possible that periods of cool-aridity were interspersed within the generally hot and arid character of the Mid-Holocene. Most records from this time period are aridity proxies. Other than isotopic analyses involving $\delta^{18}O$ and δD there are few techniques that allow for direct

means of temperature reconstruction. Warm temperatures have been assumed to accompany periods of aridity. The relationship between these parameters is strong. Nevertheless, this question will not be addressed until new techniques are brought to bear that allow for the independent assessment of these variables within a single substrate.

2.5 Conclusions

Similar to the findings of Cormie *et al.* (1994a, 1994c) for white tailed deer, the δD composition of non-exchangeable hydrogen from modern bison bone collagen demonstrates significant differences relative to location and latitude in continental North America. These differences seem to mirror the continental δD distribution of growing-season rain. This distinction remains significant even when prehistoric and modern bison from the same region (Saskatchewan) are compared to modern animals from a different one. Withstanding this, smaller yet still significantly different compositional variations exist between temporally distinct groups of bison from a single location (southern Saskatchewan). Differences in the hydrogen isotope composition of bone collagen from the prehistoric bison evaluated in this study almost certainly represent differences in climate and particularly variations in the environmental temperature within which these animals lived and their tissues formed.

A better understanding of the role that drinking versus dietary water plays, relative to the D/H composition of animal tissues, would facilitate the use of hydrogen isotope compositions of bone for the quantitative reconstruction of climate. In addition, a controlled and more detailed study of the continental-wide variation of D/H ratios amongst modern bison bone collagen, would assist regional climate reconstruction based on δD values. Finally, consideration of hydrogen values may provide a contextually linked climate proxy against which other biologically relevant isotope data from a particular individual may be evaluated. Because of their direct link to local temperature, δD values may be used to gauge the relationship which other elemental stable isotope values in bone tissue have to climate. Similarly, other forms of temporally specific site and artifact analysis may benefit from an equally specific

indicator of climate. In this way, a more complete analysis of prehistoric ecology may be possible.

Chapter 3. STABLE ISOTOPE ECOLOGY OF SOUTHERN SASKATCHEWAN BISON THROUGHOUT THE HOLOCENE

3.1 Introduction

From the end of the Pleistocene, throughout most of the Holocene, bison were a singularly important resource for the indigenous peoples of the Great Plains (Roe 1951; Bryan 1991). Bison social and foraging behaviour, at times, dictated the movements of human groups and was an important determinant of subsistence practices (Verbicky-Todd 1984; Frison 1991). Understanding the pre-contact ecology of this species is essential for archaeologists and paleoecologists engaged in Great Plains research. Nevertheless, the behavioural patterns of prehistoric bison, particularly those of the extinct forms, are poorly understood. Current models of prehistoric bison behaviour derive largely from field observations of modern herds combined with supplemental inferences from historical records (Bamforth 1988; Frison 1991; Larson *et al.* 2001; Cannon 2001). Archaeologists have tended to apply these behavioural models to bison populations from prehistoric contexts. There are indications, however, that this practice may not be entirely appropriate.

Skeletal remains recovered from archaeological and paleontological contexts that date throughout the Holocene indicate a suite of morphological differences which separate Early Holocene bison from their later Holocene counterparts (Arthur *et al.* 1975; Wilson 1978; McDonald 1981). These physical differences may have mandated differing nutritional requirements and resulted in modified foraging behaviours (Bamforth 1988; McDonald 1981; Frison 1991). As a result, bison of the distant past, may have responded to changes in forage production and distribution in fundamentally different ways than extant taxa. This is particularly important when one considers that Great Plains environments have changed dramatically throughout the Holocene (Dyck 1983; Lemmen and Vance 1999). Not only may past environments have been

substantially different from anything occurring during recent or modern times, but also bison of the past may have behaved differently under analogous circumstances.

Understanding past bison populations, and by extension the peoples dependant upon them, requires that independent behavioural models be developed from prehistoric datasets for application to prehistoric bison studies. The research presented in this paper represents an attempt to infer prehistoric bison foraging behaviour from comparative stable isotope data derived from prehistoric bison remains. Climatic contexts were qualitatively re-created using stable hydrogen and stable nitrogen isotope values to infer lifetime temperature and moisture regimes. Dietary patterns, as inferred from stable carbon isotope ratios, were interpreted with respect to the indicated environmental context. The bone samples, collected from a series of archaeological sites in southern Saskatchewan, represent a time range spanning over 9,000 years. This region, on the northern periphery of the Great Plains, has a nearly continuous record of human occupation and bison presence throughout the Holocene epoch (Linnamae *et al.* 1988).

3.2 Methods

3.2.1 Sampling and Study Area

Bone samples representing 52 individual bison were collected from eight previously excavated archaeological contexts from Southern Saskatchewan. Samples from each individual were then analyzed to determine $\delta^{13}C$, $\delta^{15}N$ and δD composition. Although, most of the sites occurred within a 50 km radius of the city of Saskatoon, the oldest locality was approximately 150 km to the southwest (Figure 3.1). Only the remains of adult bison were chosen for analysis. To assure that individual animals from each time period were sampled no more than once, only replicate elements from the same side of the body were selected. During sample preparation and combustion, an extra aliquot of CO_2 gas was collected from a specimen representing each time period and submitted to the Rafter Radiocarbon Laboratory in New Zealand for AMS radiocarbon dating. The sample from the Tschetter site appeared to have been contaminated and thus, an average of previously obtained radiometric dates is used here (Linnamae 1988). All other AMS dates were determined to be acceptable and are reported and used throughout this study (Table 3.1).

Figure 3.1 Map of Southern Saskatchewan Showing the Location of Archaeological Sites Used in This Study

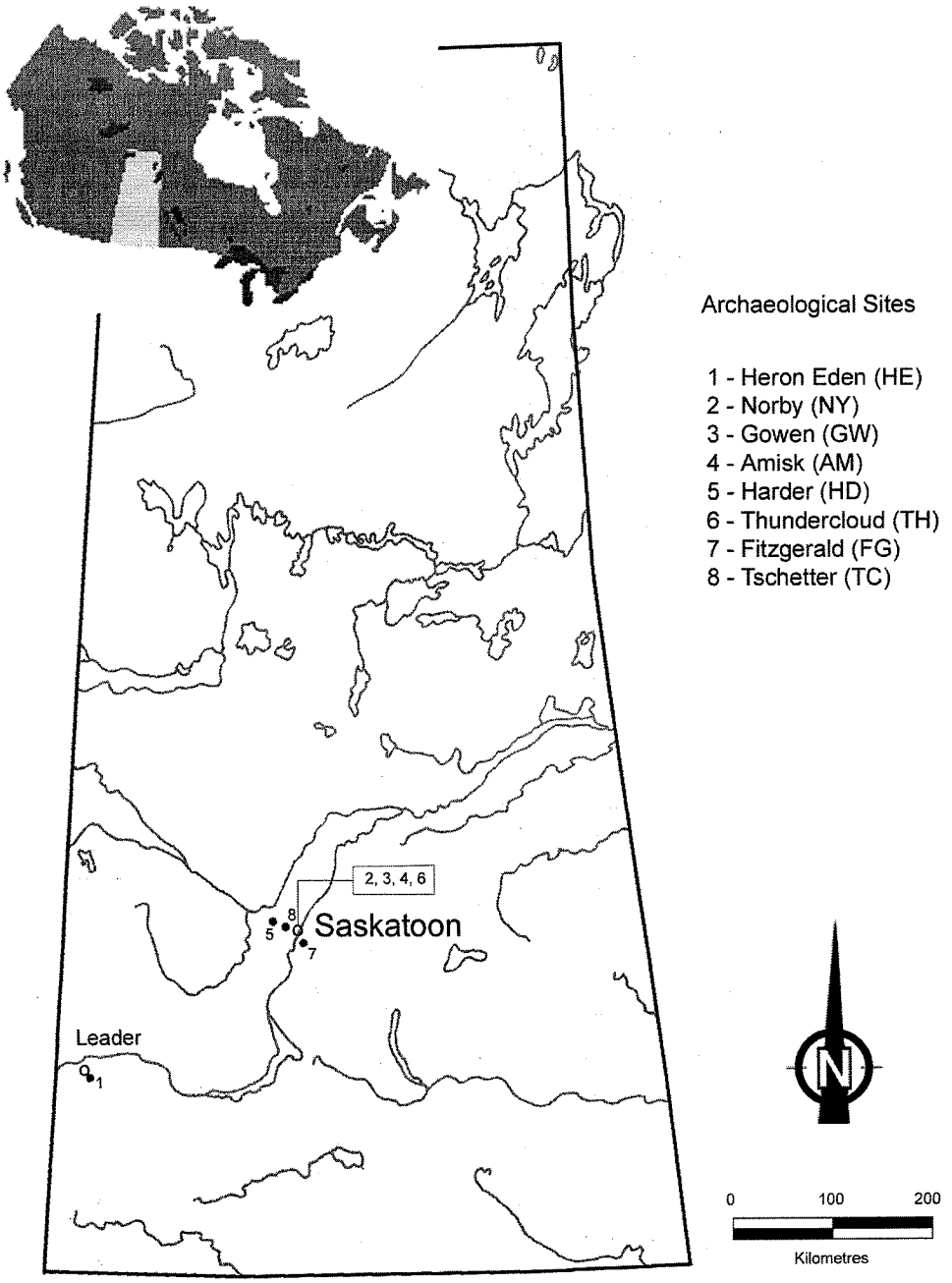


Table 3.1 Conventional and Calibrated Radiocarbon Age of Each Sampled Archaeological Component

Site	Borden Designation	Cultural Association *	^{14}C yr. BP	Yr. BP	Lab #
Heron Eden (HE)	EeOi - 11	Cody	9,168 ± 50	10,352 ± 134	NZA 15745
Norby (NY)	FaNq - 56	Mummy Cave	7,036 ± 45	7,846 ± 103	NZA 15747
Gowen (GW)	FaNq - 32	Mummy Cave	5,863 ± 55	6,653 ± 143	NZA 15746
Amisk (AM)	FbNp - 17	Oxbow (Level 4)	4,358 ± 45	4,941 ± 103	NZA 15748
Harder (HD)	FbNs - 1	Oxbow	4,221 ± 45	4,823 ± 33	NZA 15776
Thundercloud (TH)	FbNp - 25	McKean (Level 5)	3,382 ± 55	3,804 ± 13	NZA 15749
Fitzgerald (FG)	ElNp - 8	Besant	1,563 ± 45	1,442 ± 101	NZA 15750
Tschetter (TC)	FbNr - 1	Old Women's	1,035 ± 40 **		

^{14}C yr. BP, "conventional radiocarbon years before present"
Yr. BP, "calibrated radiocarbon years before present", 2 sigma range, calibrated using INTCAL98_14C, based on Stuiver *et al.* 1998
Lab #, Rafter Radiocarbon Laboratory sample number
* Cultural associations are based upon projectile point associations (see Corbeil 1995; Zurburg 1991; Walker 1992; Amundson 1986; Dyck 1977; Webster 1999; Hjermstad 1996; Linnamae 1988). For the multi-component sites (Amisk and Thundercloud) the corresponding excavation level from which the sampled bones derive is indicated
** The Tschetter date is an average of radiometric radiocarbon dates determined prior to this study (see Linnamae 1988)

3.2.2 Dietary Analysis

In settings within the continental interior, stable carbon isotope ratios ($\delta^{13}C$) in bone collagen provide an estimate of the relative consumption of C_3 and C_4 plants by an animal over the greater part of its life (Tieszen 1994). During photosynthesis these plants produce stable organic compounds for use in metabolism that are based upon either a three or four-carbon molecule (Boutton 1991b). The grasses consumed by bison exhibit a bimodal distribution of $\delta^{13}C$ values relative to their mode of photosynthesis. C_3 and C_4 species possess distinct ranges of carbon- isotope composition that differ, on average by about 14‰ (parts per thousand) (Ehleringer 1991). It is therefore, possible to calculate the relative proportion of C_3 and C_4 species that compose a particular animal's diet using a simple linear interpolation based upon the following equation (Schwarcz *et al.* 1985):

$$C_4\% = \frac{(\delta^{13}C_{measured} - \delta_3 - \Delta_{dc})}{\delta_4 - \delta_3} \times 100\%$$

where $\delta^{13}C_{measured}$ is the measured carbon isotope composition of an animal's bone collagen, δ_3 and δ_4 represent the average $\delta^{13}C$ values from within the range exhibited by most C_3 plants (-26.5‰) and most C_4 (-12.5‰) plants respectively, and Δ_{dc} represents the average trophic discrimination (5‰) between an animal's diet and its bone collagen (Tieszen 1991).

Within a specific region, the distribution of these plant species and thus, their relative availability as forage, is primarily dictated by an interaction of climate and local topography (Teeri and Stowe 1976; Boutton *et al.* 1980; Tieszen 1994). Throughout most of the year, the temperate nature of the Northern Plains conveys a competitive advantage upon C_3 plants which are physiologically adapted for optimal performance under cool environmental conditions (Tieszen *et al.* 1997a; Sage *et al.* 1999). However, warm periods, particularly during the summer months, afford an opportunity for C_4 species to become competitive. Growing seasons which are both warm and reasonably moist may result in an increase in overall C_4 distribution (Qi and Redmann 1993). Thus, providing that moisture is sufficient, the distribution and nutritional quality of both C_3

and C_4 biota will ultimately change with respect to the prevailing regional temperature regime (Sage *et al.* 1999; Long 1999).

Drought, however, induces physiological stress in most plants regardless of environmental temperature (Buchner 1980; Strahler and Strahler 1992). A lower availability of moisture results in a reduced potential for succession and a lowered nutritional quality across both C_3 and C_4 species (Long 1999; Sage *et al.* 1999). C_3 plants suffer markedly during periods of aridity regardless of ambient temperature. C_4 plants, however, possess physiological adaptations which allow them to use water more efficiently than C_3 varieties (Knapp and Medina 1999). As a result, they are somewhat drought tolerant. During hot, dry periods, C_4 plants are better able to reduce the deleterious effects of aridity upon their tissues and thus, retain a higher nutritional quality relative to C_3 species (Ozturk *et al.* 1981). This benefit is subsequently negated during cooler periods when the higher temperature requirements of C_4 plants are not met (Long 1999).

Throughout most of the year when moisture is sufficient to maintain the nutritional quality of the subsistence base, bison on the Northern Plains are reasonably unselective while grazing, but primarily consume C_3 grasses reflecting their dominance of available forage (Tieszen 1991). During arid periods when the nutritional quality of available plant tissues decrease, bison like most other ruminants, must actively select for forage of the highest available quality to meet their basic nutritional requirements (Bamforth 1988). In the late summer for example, when temperatures are high and drought conditions are most prevalent, their consumption of C_4 grasses dramatically increases to a point where their diets disproportionately reflect the availability C_4 forage (Vinton *et al.* 1993; Tieszen 1994; Steuter *et al.* 1995). However, during episodes of cool-aridity, this selectivity provides little benefit given the physiological limitations of both C_3 and C_4 species under such circumstances (Long 1999).

Thus, while the relative composition of bison diet usually reflects regional forage distributions irrespective of climate, hot/dry periods necessitate a selective response that can complicate this association. Furthermore, since the majority of C_4 grasses are consumed during a restricted period in the late summer, changes in distribution should have only a minor effect upon the relative consumption of grasses

on an annual basis. Rather, changes in the duration of the period during which most C_4 grasses are consumed will have the most substantial impact upon net annual diet. If climatic change leads to the prolongation of seasonal droughts, bison will significantly increase their consumption of C_4 grasses causing their diets to be less representative of local plant distributions. The more prolonged the drought, the greater this effect will potentially become.

In accordance with this phenomenon, climatic shifts over the lifetime of an individual which involve both a temperature increase and a moisture decrease will be expected to have an observable effect upon the lifetime consumption patterns of bison foraging in the plains of Saskatchewan. In addition, the impact of such episodes upon the plant composition of bison diet should be much more substantial than any dietary changes which could potentially occur in response to climates associated with a shift in the distribution of local vegetation. Thus, climate changes leading to an increase in drought frequency over the lifetime of an individual bison may be expected to have a significant, though indirect, effect upon the $\delta^{13}C$ composition of the animal's tissues.

3.2.3 Climate Reconstruction

In contrast with stable carbon isotope data, stable hydrogen and stable nitrogen isotope ratios in bison bone collagen should reflect a more direct relationship with climatic variables. The isotopic composition of hydrogen in the bone collagen of prehistoric bison may be used as an indicator of habitat temperature over the lifetime of an individual animal (Cormie *et al.* 1994b, 1994c). The δD composition of herbivore tissue is derived from a long-term integration of dietary water and drinking water sources (Hobson *et al.* 1999). Since the hydrogen-isotope composition of all meteoric waters available to plants and animals in a given area reflect temperature at the site of local precipitation, δD values in herbivore bone collagen should reflect the systemic influences of the local thermal regime (Gat 1996). This relationship is particularly strong in arid, continental ecosystems that have low relative humidity and where temperature mediated processes such as evaporation and evapo-transpiration primarily control hydrogen isotope discrimination in meteoric and biologic waters (Dongmann *et al.* 1974; Ehleringer and Dawson 1992; Schoetterer *et al.* 1996).

Differences in the stable nitrogen isotope composition of tissues also have the potential to address climatic phenomena. Although stable nitrogen isotope ratios may be used to examine a variety of physical and biological processes; studies which are limited to an analysis $\delta^{15}N$ variations in tissues of a single species, provide control for a variety of effects which might result from the differing metabolic adaptations of different taxa (Cormie and Schwarcz 1994). Under such restrictions, variances in the stable nitrogen isotope value of bone collagen from different individuals may potentially be used as a proxy for habitat-aridity. Several studies have proposed a link between local climate and the nitrogen isotope composition of soils and the tissues of plants and animals (Heaton *et al.* 1986; Ambrose and DeNiro 1989; Fizet *et al.* 1995). Although there is a strong negative correlation between annual rainfall and $\delta^{15}N$ values in herbivore bone collagen, only a small part of this relationship can be explained by differences in the isotopic composition of dietary plants (Ambrose 1991). Instead, "physiological adaptations to water stress and/or low protein diets most likely account for the majority of $\delta^{15}N$ enrichment observed in arid-land herbivores" (Pate *et al.* 1998:44).

In times of water stress, some animals possess metabolic adaptations which promote an increase in the urea content of their urine. This process results in the preferential excretion of ^{14}N in the increasingly concentrated urine and subsequently causes an elevation of $\delta^{15}N$ values the animal's tissues (Ambrose 1991). Alternatively, protein stress may also result in an increase of tissue values (Tieszen 1994; Fizet *et al.* 1995). Among herbivores like bison, the nutritional value of forage is primarily determined by the availability of moisture for local grasses. Regional moisture deficiency effects plant growth and ultimately reduces protein levels in the available vegetation (Ozturk *et al.* 1981; Heckathorn *et al.* 1999). Mammals consuming diets low in protein may more intensively recycle the nitrogen in their own tissues resulting in subsequent isotopic enrichment (Ambrose 1991, Hobson *et al.* 1993).

This process may be further related to water-stress mechanisms insomuch that insufficient protein intake can result in the reutilization of an animal's bodily tissue reserves which, through increased urea excretion, are already enriched (Tieszen 1994; Cormie and Schwarcz 1996; Katzenberg 2000). Unfortunately, the interrelation and

complexity of water and protein-stress mechanisms, as well as the extent to which they may be applicable to different species, remains unknown (Ambrose 2000). Nevertheless, it does seem that regional levels of aridity do exert at least an indirect influence upon $\delta^{15}N$ values in the bone collagen of terrestrial herbivores in arid ecosystems. Since this effect appears to be predictable, $\delta^{15}N$ values in bone may at the least be used to provide a subdued qualitative measure of habitat moisture during the lifetime of the source animal.

3.2.4 Sample Preparation and Analysis

Bone samples weighing about three grams were selected from each specimen for stable isotope analysis. All samples were then inspected, mechanically cleaned and washed in a hydrosonic bath to remove dirt and other particulate matter. Bone collagen was then extracted and purified following the methods described by Sealy (1986). Briefly, all samples were de-mineralized in 0.25 M HCl at room temperature over a period of a several weeks. Each was then rinsed to a neutral pH using de-ionized water and treated with 0.125 M NaOH at room temperature for 20 hours to remove humic acids. Next, all specimens were again rinsed to neutral pH using de-ionized water, frozen and finally freeze-dried. Prior to combustion, each was ground and homogenized using an analytical mill. Due to the complication of hydrogen exchange, combustion and measurement of δD values was performed separately from that of $\delta^{13}C$ and $\delta^{15}N$ values. Additional preparation was required.

A portion of the total hydrogen in bone collagen is available for isotopic exchange with ambient water vapour (DeNiro and Epstein 1981a; Wassenaar and Hobson 2000). It was therefore, necessary to quantify and eliminate the effect of this uncontrolled, temperature-dependant variable. The proportion of exchangeable hydrogen in bone collagen was first determined using a static equilibration technique (see Wassenaar and Hobson 2000) on samples of modern Saskatchewan bison bone collagen (see Chapter 2). Each specimen was equilibrated for two hours at a constant temperature (135°C) with steam of differing hydrogen isotope composition (-135‰, +115‰, +525‰) and then measured to determine the resulting D/H ratio. The hydrogen isotope composition of each sample, both prior to equilibration and after each trial with

a different steam, was compared and subsequently used to calculate the proportion of exchangeable hydrogen in each modern collagen sample. The average percentage of hydrogen available for isotopic exchange at this temperature was determined to be 20.0 ± 2.5 (n = 4). This proportion is consistent with other published estimates for various biological tissues (Schoeller *et al.* 1986; Cormie *et al.* 1994b; Hobson *et al.* 1999). Subsequently, all prehistoric samples were standardized by equilibration with steam of known δD value (δD = -135‰) at 135°C for two hours and calibrated according to the determined portion of exchangeable hydrogen (see Wassenaar and Hobson 2000).

After equilibration in Vycor breakseal tubes, all water vapour was removed cryogenically. Samples were sealed under a vacuum and combusted at 850°C in the presence of cupric oxide, followed by cryogenic separation of CO_2 and N_2 from H_2O. Waters of combustion were reduced to H_2 gas by using hot zinc, and D/H ratios were measured on a Micromass Optima dual inlet isotope-ratio mass spectrometer. Stable hydrogen isotope results are reported using delta (δ) notation in parts per thousand (‰) deviation from the Vienna Standard Mean Ocean Water (VSMOW) standard.

$\delta^{13}C$ and $\delta^{15}N$ values were determined from un-equilibrated samples of bone collagen. Approximately 1.5 mg of each specimen was weighed into tin capsules and introduced into an automated sample manifold. Sample combustion and the measurement of stable carbon and nitrogen isotope ratios were subsequently performed using an elemental analyzer coupled to a Micromass Optima dual inlet isotope ratio mass spectrometer that was re-configured for continuous flow. Stable carbon isotope results are reported using delta (δ) notation in parts per thousand (‰) deviation from the PeeDee Belemnite (PDB) standard. Stable nitrogen isotope results are reported using delta (δ) notation in parts per thousand (‰) deviation from the atmospheric nitrogen gas (AIR) standard.

3.3 Results

3.3.1 Collagen Preservation

Although somewhat variable, the quality of collagen extracted from the bison bone samples was determined to be generally good (see Appendix D). Specimens that yielded collagen in amounts equivalent to, or greater than 5% of the total dry,

unprocessed mass of the original sample, were considered to be acceptable (Schoeninger *et al.* 1989). C/N ratios within a range of 2.9 and 3.6 were considered to be characteristic of unmodified collagen protein (DeNiro 1985). 37 of the 52 sampled archaeological specimens met these criteria and following extraction produced solid, translucent collagen pseudomorphs.

The remaining fifteen samples comprised the bone assemblages from the Norby and Harder sites. Although these specimens possessed acceptable C/N ratios, they yielded very low concentrations of collagen (< 5%). The insoluble residues extracted from these samples were of a more liquid consistency and in most cases a small portion of the resulting collagen was lost during subsequent filtration. The resulting yields were thus artificially reduced. In addition, many of the Harder specimens contained an insoluble mineral component (determined by x-ray diffraction to be quartz silica) that was subsequently removed from the resulting collagen during filtration. The inclusion of this material in the initial mass assessment of each unprocessed dry sample and its subsequent removal, would have obscured the calculated collagen yields. However, even with low yields, all of these samples provided sufficient gas volumes upon combustion to analyze for stable isotope ratios. These results were subsequently included in the following analysis.

3.3.2 The Isotopic Composition of Collagen Samples

The isotope data developed through this study (Table 3.2) were analyzed by a series of simple linear regressions comparing the δD, $\delta^{15}N$ and $\delta^{13}C$ values of each sample (Figure 3.2). There was found to be no significant relationship between the $\delta^{15}N$ composition of the collagen samples and either δD ($r^2 = 0.006$, $F_{1,50} = 0.284$, $P<0.60$) or $\delta^{13}C$ values ($r^2 = 0.002$, $F_{1,50} = 0.105$, $P<0.80$). However, a comparison of the δD and $\delta^{13}C$ composition of all samples did suggest a weak ($r^2 = 0.21$) but significant ($F_{1,50} = 13.105$, $P<0.01$) correlation between these criteria. A statistical comparison of each time period was also undertaken in an attempt to identify important temporal differences amongst these variables.

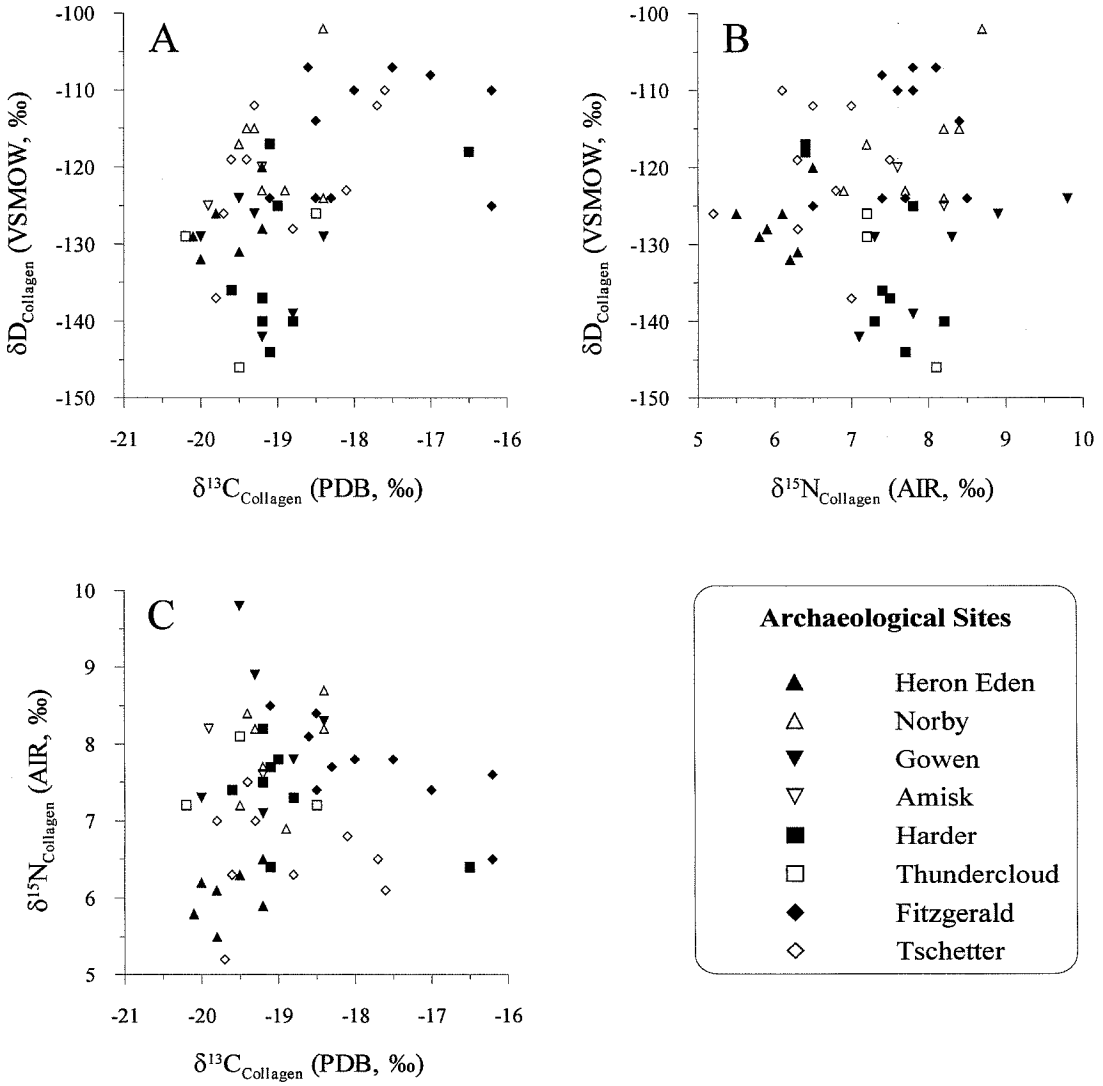
The hydrogen isotope composition of the individual bison varied from between -146‰ and -102‰ with a maximum difference of 44‰. Means for each of the time

Table 3.2 Isotopic Composition of Bone Collagen Samples of Individual Bison From Prehistoric Archaeological Sites in Southern Saskatchewan

Site	n	Species *	Element **	Collagen			
				δD (‰)	$\delta^{15}N$ (‰)	$\delta^{13}C$ (‰)	% C_4
HE	7	*B. antiquus*	R. metacarpal	-127 ± 4.0^{ab}	6.0 ± 0.3^{a}	-19.7 ± 0.4^{a}	13.2 ± 2.6
NY	7	*B. occidentalis*	L. metacarpal	-117 ± 7.7^{a}	7.9 ± 0.7^{b}	-19.0 ± 0.5^{ab}	17.8 ± 3.3
GW	6	*Bison sp.*	R. astragalus	-132 ± 7.3^{b}	8.2 ± 1.0^{b}	-19.2 ± 0.6^{a}	16.4 ± 4.0
AM	2	*B. bison*	R. scaphoid	-123 ± 3.5^{ab}	7.9 ± 0.4^{bc}	-19.6 ± 0.5^{ab}	13.9 ± 3.5
HD	8	*B. bison*	L. tibia	-132 ± 10.6^{b}	7.3 ± 0.6^{bc}	-18.8 ± 1.0^{ab}	19.2 ± 6.9
TH	3	*B. bison*	R. metatarsal	-134 ± 10.8^{bc}	7.5 ± 0.5^{bc}	-19.4 ± 0.9^{ab}	15.0 ± 6.1
FG	10	*B. bison*	L. metatarsal	-115 ± 8.0^{ac}	7.7 ± 0.6^{b}	-17.8 ± 1.0^{b}	26.5 ± 7.3
TC	9	*B. bison*	L. metatarsal	-121 ± 8.8^{ab}	6.5 ± 0.7^{ac}	-18.9 ± 0.9^{ab}	18.7 ± 6.3

Means within a column followed by different letters are significantly different from each other (ANOVA, Tukey's test, $P \leq 0.05$)
Bison sp., Indeterminate species
* Species determinations are based upon previous studies (see Corbeil 1995; Walker 1979, 1992; Amundson 1986; Dyck 1977; Webster 1999; Hjermstad 1996)
** Refers to the specific element used to avoid replicate sampling of individuals from each site; R. "right", L. "left"

Figure 3.2 Linear Regressions of δD, $\delta^{13}C$ and $\delta^{15}N$ Values From Bison Bone Collagen Samples



A - δD vs. $\delta^{13}C$, $r^2 = 0.21$, $F_{1,50} = 13.105$, $P<0.01$

B - δD vs. $\delta^{15}N$, $r^2 = 0.006$, $F_{1,50} = 0.284$, $P<0.60$

C - $\delta^{15}N$ vs. $\delta^{13}C$, $r^2 = 0.002$, $F_{1,50} = 0.105$, $P<0.80$

periods ranged over approximately 19‰ from averages of -134‰ to -115‰. Significant differences were found to exist between at least two of these groups (ANOVA, $F = 5.204$, $df = 7,44$, $P < 0.001$) and a multiple comparison analysis (Tukey's HSD test) was used to identify where these differences occurred. Bone from the Fitzgerald site population had the most positive mean (-115 ±8.0‰) and differed significantly from the Gowen ((-132 ±7.3‰) $P < 0.01$), Harder ((-132 ±10.6‰) $P < 0.01$) and Thundercloud ((-134 ±10.8‰) $P < 0.05$) site populations. Bison from the Norby site also had significantly higher mean δD values (-117 ±7.7‰) than individuals from both the Gowen ($P < 0.05$) and Harder sites ($P < 0.05$).

The $\delta^{13}C$ values of the prehistoric bison bone collagen samples range over approximately 4‰ from -20.2‰ to -16.2‰. Correspondingly, the calculated percentage of C_4 plants composing the diet of the individual bison exhibited a total variation of about 28% from a low of 10% to a high of 38.2%. The mean values from each time period were spread over a range of about 2‰ from -19.7‰ to -17.7‰. A statistical analysis suggested that at least one of these groups differed significantly from the others (ANOVA, $F = 4.272$, $df = 7,44$, $P < 0.001$). For the most part, mean calculations from each temporally distinct group indicated that C_4 plants formed between roughly 13% and 19% of total diet. The one exception to this was the Fitzgerald site population. The average proportion of C_4 plants in the diet of bison from this time period was calculated to be roughly 26.5%. A multiple comparison analysis (Tukey's HSD) of the $\delta^{13}C$ values within each group demonstrated a statistical difference between the consumption patterns of the Fitzgerald site bison (-17.8 ±1.0‰) and those of the Heron Eden ((-19.7 ±0.4‰) $P < 0.01$) and Gowen ((-19.2 ±0.6‰) $P < 0.05$) site populations.

Variation in the stable nitrogen isotope composition of the collagen samples occurred across a range of about 4.6‰. The lowest measured $\delta^{15}N$ value was 5.2‰ while the most positive was 9.8‰. Significant differences were found to exist between at least two of the temporally distinct groups (ANOVA, $F = 8.972$, $df = 7,44$, $P < 0.001$). The mean values for each population varied from a low value of 6‰ to a high of 8.2‰ over a 2.2‰ distribution. A multiple comparison analysis (Tukey's HSD) demonstrated that the mean nitrogen isotope composition of the Heron Eden bison (6.0 ±0.3‰) population, which was the least positive of all groups, differed most

significantly ($P < 0.01$) from the Norby (7.9 ±0.7‰), Gowen (8.2 ±1.0‰), Harder (7.3 ±0.6‰) and Fitzgerald (7.7 ±0.6‰) populations and to a lesser extent ($P < 0.05$) from the Amisk (7.9 ±0.4‰) and Thundercloud (7.5 ±0.5‰) groups. Bison from the Tschetter site represented the only group from which the Heron Eden bison did not significantly deviate. In turn, the average $\delta^{15}N$ composition of the Tschetter site bison bone (6.5 ±0.7‰) was found to differ substantially ($P < 0.01$) from that of the Norby, Gowen and Fitzgerald bison groups.

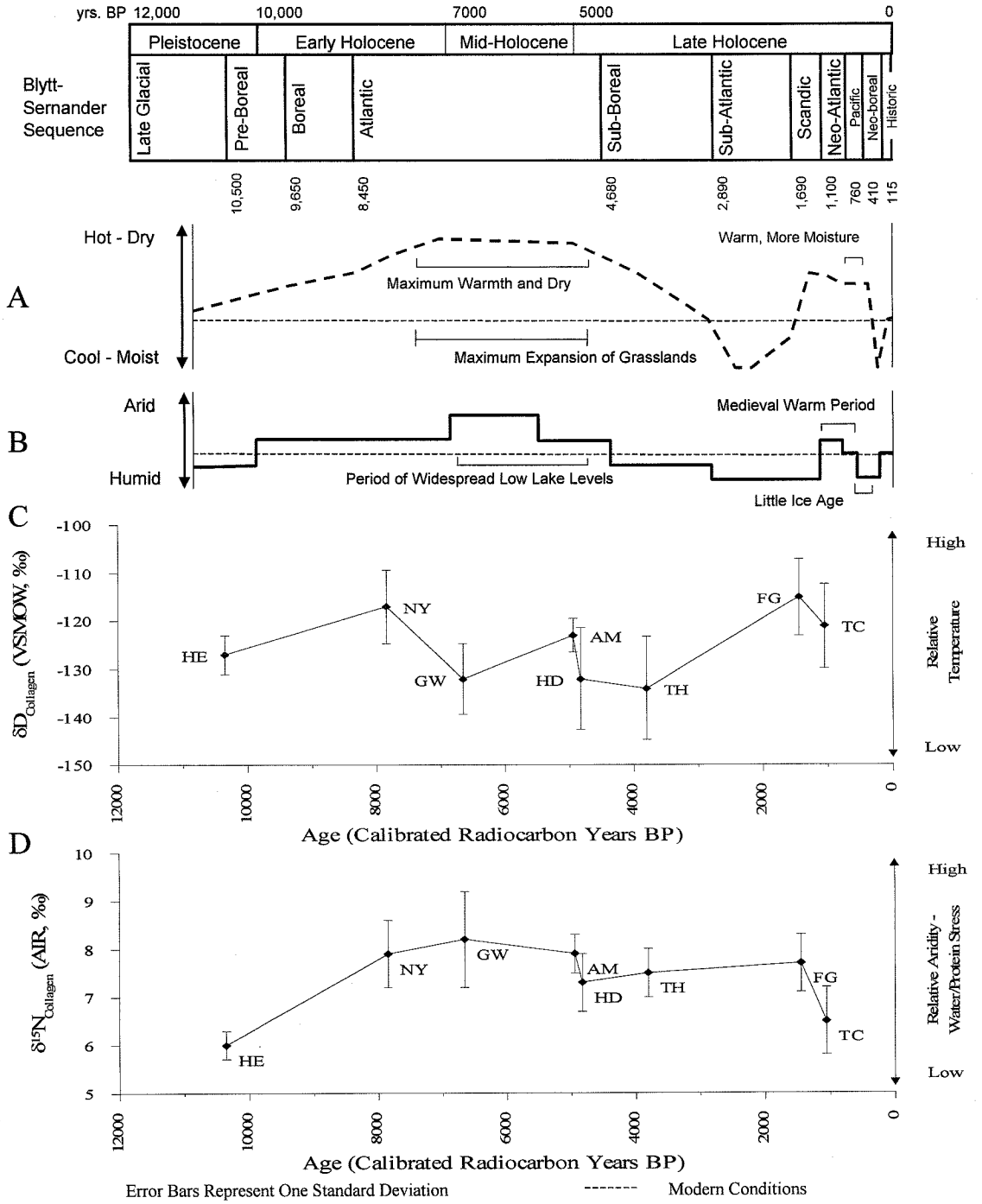
3.4 Discussion

3.4.1 Paleoclimatic Implications

Although δD and $\delta^{15}N$ values in the bone collagen of the sampled bison vary significantly through time, no direct correlation is evident between the two (see Figure 3.2). This is not unexpected given the assumption that each relates to a different aspect of local climate during the lifetimes of the sampled individuals. While environmental aridity is often positively correlated with ambient temperature, this is not always the case. As a result, climatic reconstruction based upon stable isotope data may be difficult to reconcile with comparable interpretations deriving from different paleoclimate proxies. Other paleoecological markers may be of insufficient resolution to distinguish evidence of temperature change from that of moisture variation (Vance *et al.* 1995). In addition, while isotopic ratios in bone collagen represent a finite time range within the lifetime of an individual animal, other proxies are commonly resolved to a scale of decades or even centuries (Sauchyn 1997; Lemmen and Vance 1999). Nevertheless, the climatic reconstructions presented here are surprisingly consistent with other general models of Holocene climate changes in southern Saskatchewan (Figure 3.3). $\delta^{15}N$ and δD measurements from the bone collagen of prehistoric bison present a plausible sequence of Holocene temperature and aridity shifts.

The Heron Eden site, although geographically distinct from the other analyzed archaeological contexts, provides a window on climate during the Pleistocene/Holocene transition (~10,500 - 8,500 yr. BP). The landscapes of this period, although poorly understood, were probably subject to a warming and drying trend relative to the end of regional glaciation (Dyck 1983; Lemmen and Vance 1999). This pattern induced the

Figure 3.3 Holocene Climate Changes in Southern Saskatchewan: A Comparison of Conventional Proxies with Temperature and Aridity Models Developed From the δD and $\delta^{15}N$ Composition of Bison Bone Collagen



A - adapted from Dyck 1983; B - adapted from Lemmen and Vance 1999
C, D - From Data Developed During This Study (2003)

rapid transformation of local spruce forest into grassland (Sauchyn 1997; Yansa and Basinger 1999). Nevertheless, moderate temperatures and significant levels of moisture would have characterized the region toward the beginning of this episode. At 10,300 yr. BP, bone collagen from the Heron Eden site bison possesses an average stable nitrogen isotope composition that is significantly more negative than six of the other seven sampled populations. δD measurements were intermediate. Together, these data suggest the occurrence of a moist and moderately temperate climate. Interestingly, the modern setting of the Heron Eden site is substantially more xeric than the Saskatoon area (Corbeil 1995; Acton *et al.* 1998). Given the transgressive nature of climate change and grassland expansion, it is possible that at 10,300 yr. BP, Saskatoon was significantly cooler and wetter than at present.

Following this period and throughout the subsequent establishment of the grasslands, it is widely believed that the Northern Plains became significantly more arid (Dyck 1983; Vance *et al.* 1995; Lemmen and Vance 1999). The $\delta^{15}N$ data presented here lend some support for this interpretation. Of all time periods, the most positive average results come from the three archaeological contexts that date to between 7,900 and 4,900 years BP. This time frame is roughly concurrent with the onset of a climatic event commonly referred to as the "Mid-Holocene Climatic Optimum" or "Altithermal" during which the dry and potentially warm conditions of the Atlantic Climate Episode (~8,500 - 4,600 BP) reached a maximum. Although this event was of significant enough duration to allow the plains to expand beyond their modern boundaries to the north and east, the increasingly xeric conditions may have resulted in a substantial desiccation of regional vegetation (Dyck 1983; Reeves 1973; Vance *et al.* 1995; Vreeken 1999).

Proxy data from a variety of studies testify to the presence of moisture deficiency throughout the Saskatchewan plains during the Mid-Holocene (Sauchyn and Sauchyn 1991; Sauchyn 1997; Lemmen and Vance 1999; Birks and Remenda 1999; Richmond and Goldsborough 1999; Yansa and Basinger 1999). Low lake levels and an increasing frequency of fire are documented at several sites across the region. Nevertheless, there is little evidence to directly characterize the corresponding temperature regime despite the widespread assumption of pervasive regional warmth. Although δD measurements from the Norby (~7,800 yr. BP) and Amisk site (~4,900 yr.

BP) bison populations would be consistent with a warmer climate, values from the Gowen site (~6,700 yr. BP) bison are substantially more negative.

The Gowen data contrast significantly with those of the Norby site and when considered alongside the corresponding $\delta^{15}N$ results, depict a climate that is moderately temperate, but notably arid. Cool, dry weather patterns are characteristic of temperate deserts and are largely detrimental to plant species that lack specific adaptations for these environments (Strahler and Strahler 1992; Long 1999). Such periods, occurring within the context of a prolonged Mid-Holocene drought, may have had a substantial impact on carrying capacities across southern Saskatchewan. Thus, while the information from the Gowen site population is consistent with the interpretation of the Middle Holocene as largely arid, it also suggests that the period may have been characterized by a degree of temperature variability.

The ending of the Atlantic Climate Episode apparently brought ameliorating weather conditions (Dyck 1983; Vance *et al.* 1995; Lemmen and Vance 1999). δD and $\delta^{15}N$ values from the Harder bison population (~4,800 yr. BP) would seem to indicate a rapid transition. The average stable hydrogen isotope composition of bone collagen from the Harder bison is similar to that of the Gowen site population, denoting the onset of cooler average environmental temperatures. Nevertheless, $\delta^{15}N$ measurements indicate only a marginal increase in average moisture and thus, despite ameliorating temperatures, the continuation of moderate aridity. δD and $\delta^{15}N$ measurements from the Thundercloud bison population suggest the presence of a largely similar climate at about 3,800 yrs. BP. However, the high degree of δD variability associated with the three Thundercloud specimens is somewhat conspicuous. There is a possibility that the Thundercloud assemblage is composed of more than one temporal event (Webster 2002, personal communication). Nevertheless, data from the Harder and Thundercloud bison seem to be consistent with the interpretation of a cooling trend during the early part of the Late Holocene. This pattern is thought to have peaked between about 3,000-2,000 years BP, resulting in a much cooler and moister climate than occurs at present (Dyck 1983; Vickers 1986). Unfortunately, this project presents no specific data for this time period.

By about 1,450 years BP, the habitat of the Fitzgerald bison had apparently returned to conditions similar to those of the early part of the Middle Holocene. The average δD and $\delta^{15}N$ bone collagen measurements from the Fitzgerald bison population were remarkably close to those of the Norby bison and indicate a significant shift in temperature from the Amisk and Thundercloud time periods. The Fitzgerald assemblage has been dated to within a climatic episode known as the Scandic (~1,700 - 1,100 BP) (Dyck 1983). Although not as prolonged in duration as the Atlantic period, Scandic environments have been similarly described to be hot and dry (Dyck 1983; Lemmen and Vance 1999). While the occurrence of this event within southern Saskatchewan appears to have been somewhat variable (Frison and Mainfort 1996; Sauchyn 1997; Shang and Last 1999; Richmond and Goldsborough 1999), sedimentary records from Redberry Lake near Saskatoon provide evidence of a localized moisture deficiency between about 2,500 and 1,500 years BP (Van Stempvoort *et al.* 1993). During this period, it is entirely possible that local vegetation experienced distribution and nutritional changes of a similar nature, though not necessarily magnitude, to those hypothesized to have occurred during the droughts of the Middle Holocene.

Much of the northern hemisphere is thought to have remained continuously warm from the onset of Scandic climate at about 1,700 BP until the end of the subsequent Neo-Atlantic phase (~1,100 - 800 yr. BP) (Dyck 1983; Van Stempvoort *et al.* 1993). However, while arid conditions continued throughout Europe, the onset of the Neo-Atlantic is thought to have engendered a significantly more humid climate in North America (Reeves 1969; Dyck 1983; Vickers 1986; Van Stempvoort 1993; Sauchyn and Beaudoin 1998). Though somewhat short-lived, this warm-moist period probably included growing-seasons that were favorable to warm-season grasses and thus of potential benefit to resident bison populations (Greiser 1994). The isotopic data derived from the Tschetter site bison would seem to support the existence of such a climatic regime at about 1,050 years BP. Reasonably positive bone collagen δD values testify to the persistence of warm ambient temperatures, while $\delta^{15}N$ measurements indicate a significant moisture regime similar to that occurring at the Heron Eden site during the Pleistocene/Holocene transition.

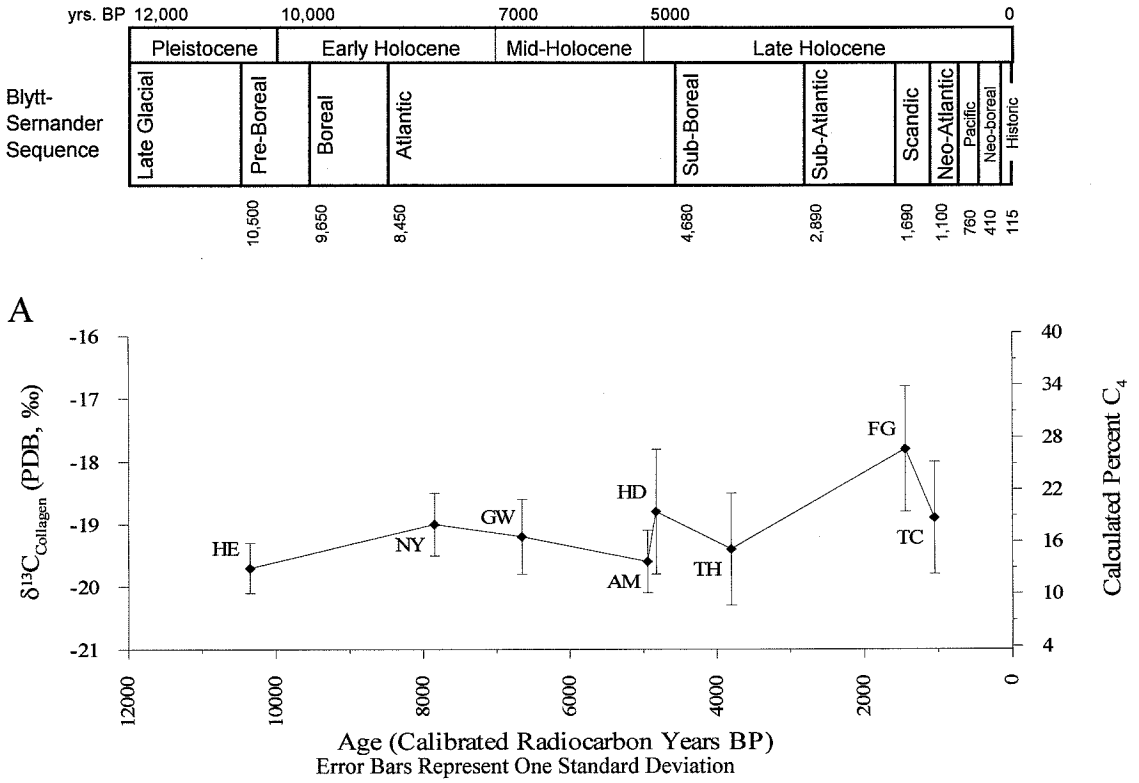
3.4.2 Paleodietary Implications

An analysis of the stable carbon isotope composition of bone collagen samples from Saskatchewan bison has demonstrated that dietary patterns throughout the Holocene were largely indicative of forage consumption within a C_3-dominated community (see Table 3.2). On average, C_3 species were found to account for 81.3% (-18.9‰) of bison diet, very close to modern estimates of overall C_3 forage production (~80%) within this region (Coupland 1961; Tieszen 1994; Tieszen *et al.* 1997a; Sage *et al.* 1999; Larson *et al.* 2001). Since the end of the Pleistocene southern Saskatchewan and the larger area of the Canadian Plains has been a temperate region dominated by the occurrence of C_3 grass species (Dyck 1983; Tieszen *et al.* 1997a; Clark *et al.* 2001). Thus, in a very general way, the diets of all of the sampled bison, regardless of temporal context, reflect the forage composition of their regional habitat.

Nevertheless, a variety of studies based upon different paleoecological proxies have shown that regional climate and vegetation distributions in the northern plains have fluctuated substantially over the last 10,000 years (Sauchyn 1997; Lemmen and Vance 1999; Clark *et al.* 2001). Similarly, δD and $\delta^{15}N$ values determined from the bison bone collagen samples analyzed for this project indicate significant climatic differences among the sampled time periods. It is reasonable to assume that the characteristics of local forage communities would have varied in response to these changing climatic patterns. Nevertheless, the measured $\delta^{13}C$ value of bison bone collagen appears to have remained reasonably stable throughout the Holocene (Figure 3.4). Bison diets from each time period do not appear to reflect the changes in forage distribution which may be expected in response to the fluctuation of regional climate.

Some of this discrepancy may result from physiological and geochemical mechanisms which alter the carbon isotope composition of meteoric substrates prior to their ingestion by bison. During hot and/or dry periods, aridity mediated alterations to the photosynthetic process employed by C_3 plants can result in a positive increase in the $\delta^{13}C$ value of their tissues (Ehleringer 1991; Tieszen 1994). It is thus possible that increases in bison bone collagen $\delta^{13}C$ may result from the steady consumption of C_3 plant tissues with an increasingly positive $\delta^{13}C$ composition. A similar effect may also act to modify the δD composition of C_3 plant tissues, making them grow slightly more

Figure 3.4 The Plant Composition of Bison Diet as Inferred From $\delta^{13}C$ Values in the Bone Collagen of Prehistoric Animals



A - From Data Developed During This Study (2003)

C_4 Percentages Calculated From the $\delta^{13}C$ data (see Schwarcz *et al.* 1985)

positive in arid contexts. Unfortunately, these processes alone do not account for the full range of climatic or dietary variation revealed in the current study.

Bison are known to change their dietary patterns in response to a variety of biotic and abiotic criteria (Moodie and Ray 1976; Singer and Norland 1994; Hart 2001; Fortin *et al.* 1997). On a regional scale, forage distribution is probably the most important determinant of bison subsistence (Vinton *et al.* 1993; Steuter *et al.* 1995; Fortin *et al.* 2002). Like most large ruminants, bison can tolerate a fairly low quality diet providing that adequate overall volumes of forage are available (Peden *et al.* 1974; Hanson 1984; Bamforth 1988). The southern Saskatchewan plains are dominated by C_3 grasses. Throughout most of the year this foliage is of sufficient nutritional value toallow bison to forage in a reasonably indiscriminate fashion and as a result, their overall diets come to reflect local plant distributions. Quality becomes an important issue at local or seasonal scales (Vinton *et al.* 1993; Steuter *et al.* 1995; Fortin *et al.* 2002). In specific instances when plant distributions change or when the nutritional value of local vegetation is reduced below their tolerances, bison must compensate by adjusting their range and/or actively selecting for plants of higher nutritional quality (Bamforth 1988; Heckathorn *et al.* 1999). This behaviour may help to explain the temporal stability of the bone collagen $\delta^{13}C$ values observed among the prehistoric bison examined in this study.

Bison, like most large ruminants, primarily respond to changes in local forage distribution by altering their movement patterns (Bamforth 1988). As a result of seasonal differences in quality, they prefer C_3 forage in the fall, winter and spring; while selecting C_4 forage in the summer (Steuter *et al.* 1995). Their movements are largely designed to help them acquire the appropriate amount of seasonally adequate forage, regardless of local distribution. Thus, under most climatic conditions, the average annual composition of their diets should remain stable, reflecting only the greater regional abundance of C_3 and C_4 species.

The absence of any strong correlation between $\delta^{13}C$ values in bison bone collagen and either the δD or $\delta^{15}N$ composition of the same substrate provides further evidence for this conclusion. Despite the fact that bone collagen δD and $\delta^{15}N$ values indicate a series of substantial climatic changes; bison during most of the prehistoric

time periods likely adjusted their foraging behaviour in response to the changing vegetation patterns. As a result, their diet remained constant, regardless of climate driven changes in forage distribution. Although there was a weak ($r^2 = 0.21$), but significant ($p<0.01$) correlation between the δD and $\delta^{13}C$ measurements, this finding does not contradict such an interpretation. That a significant relationship exists between the two datasets is not surprising given that both hydrogen and carbon isotope values in meteoric substrates are highly correlated with environmental temperature (Hoefs 1973). Instead, it is the weakness of this correlation that is conspicuous. Quite likely a selective behaviour, altering the relative consumption of C_3/C_4 plants by bison in the different time periods, is responsible for weakening the association between the carbon and hydrogen isotope data.

Such behaviour is only viable, however, if a source of nutritionally adequate forage exists. Under sufficient moisture conditions, the seasonal cycles of C_3 and C_4 grasses provide bison with an abundant yearly supply of acceptable food. During moisture deficient periods, however, forage quality suffers. It is at these times that bison, like most other ruminants, must actively select for forage of the highest available quality (Bamforth 1988). Throughout hot, dry periods in the Northern Plains, bison may have taken advantage of the physiological specializations of C_4 plants (Heckathorn *et al.* 1999). During the late summer, for example, bison will dramatically increase their consumption of C_4 grasses which, as a result of their proclivity for warm temperatures and tolerance of drought, become nutritionally attractive. As moisture deficient periods increase in duration, bison diets will continue to reflect an increasing consumption of C_4 species. This selective response, prevalent amongst modern bison, is an essential adaptation to the seasonal cycles of the Great Plains (Bamforth 1988).

Of the prehistoric bison populations examined through the course of this study, only one demonstrated a statistically significant difference in average C_4 consumption. Stable carbon isotope values from the Fitzgerald site (~1,450 BP) bison indicate that C_4 grasses accounted for an average of 26.5% of total dietary intake. This is about 7% higher than the next highest group and about 8% higher than the average for all of the time periods (18.7%). This increased reliance upon C_4 forage is probably a result of climatic influences on the local subsistence base. δD and $\delta^{15}N$ measurements from the

same specimens indicate the simultaneous occurrence of a hot and reasonably arid climatic regime. This interpretation is consistent with other proxy reconstructions of contemporaneous Scandic period climates in southern Saskatchewan.

The physiology and behavioural responses of modern plains bison are extremely well adapted to the cycles of the Great Plains region (Steuter *et al.* 1995). Anatomically modern bison appear consistently in the archaeological record by 5,000 BP and thus, populations more recent than this are expected to demonstrate similar adaptations and tendencies (Bamforth 1988; Wilson 1978). Nevertheless, archaeological evidence has documented a variety of morphological and demographic differences in Early and Middle Holocene fossil bison populations (Guthrie 1966, 1970; Wilson 1969, 1974; McDonald 1981). It has been speculated that these earlier taxa may have exhibited behavioural differences (McDonald 1981; Bamforth 1988; Frison 1991). The evolution of the species throughout the Holocene is thought to have occurred largely as a result of pressures to adapt to expanding grassland environments (Arthur *et al.*1975; Wilson 1978; McDonald 1981). It is therefore, reasonable to conclude that many modern behaviours, beneficial within this context, similarly evolved over this period.

Bison from the Early Holocene Norby site assemblage, appear to demonstrate a dietary pattern that is inconsistent with the consumption practices evident among the Late Holocene populations. Despite δD and $\delta^{15}N$ bone collagen measurements indicating the occurrence of a hot, dry climate remarkably similar to that of the Fitzgerald period, the Norby bison do not appear to have altered their foraging behaviour to take advantage of the available C_4 biota. While it is possible that grasslands of the Early Holocene were fundamentally different than modern ones, it is more probable that this discrepancy results from a behavioural difference. During this period of specific environmental stress, the failure of the Norby bison to supplement their diets with the most nutritious forage available would have been significantly detrimental. Furthermore, if this practice was found to be a characteristic of Early Holocene bison populations it would indicate an important behavioural difference distinguishing these bison from their later Holocene counterparts.

Of all the sampled populations, bison from the Gowen time period appear to have endured the most severe environmental conditions. Bone collagen δD and $\delta^{15}N$

measurements suggest the presence of a cool, arid climatic regime. Under such conditions, the quality of almost all available plant species would have suffered and therefore, discriminate forage selection would be of little benefit (Long 1999). The most logical response for bison under such conditions would have been a dramatic increase in foraging range (Heckathorn *et al.* 1999). This is another strategy common to large ruminant herbivores in times of dietary stress (Bamforth 1988). To compensate for the lower average nutritional quality of available forage, they attempt to consume larger overall amounts of food while simultaneously searching for better pastures (Bamforth 1988; Heckathorn *et al.* 1999). Since selection is of little benefit, their diets continue to reflect regional distributions. Interestingly, the inability of the Norby bison to exploit C_4 grasses during a subsequent period of hot aridity would have necessitated a similar response. It appears that despite evident differences in climate, data from both the Norby and Gowen sites support the interpretation of the Middle Holocene as a period of significant stresses for regional bison populations.

3.5 Conclusions

The combined usage of stable hydrogen, nitrogen and carbon isotope analyses appears to have the potential to provide ecological information of a targeted, yet interrelated nature from the fossil remains of animals. Nevertheless, the regular application of isotopic methods to archaeological and paloecological problems involving bison will necessitate that specific concerns be adequately addressed. Refinements to the hydrogen isotope technique must include a quantification of the influence which drinking water has on the D/H composition of animal tissues. As well, the exact mechanisms by which aridity influences the nitrogen isotope composition of mammalian tissues must be identified and explained. Although a substantial amount of work has been dedicated to the investigation of carbon isotope ratios in the fossil remains of bison, researchers may have to pay closer attention to temporal and geographic context. The relationships between environment and behaviour may be quite variable across the landscape and also through time.

Nevertheless, there are several specific conclusions which may be drawn from the data presented in this study:

1. Hydrogen and nitrogen-stable isotope ratios in the bone collagen of adult bison may provide reasonable qualitative estimates of lifetime climatic temperature and precipitation regimes.
2. Non-isotopic proxy records of past climate change may be of an insufficient temporal resolution to allow for accurate comparative interpretations involving stable isotope data derived from the bone collagen of fossil bison.
3. The relative contribution of C_3 and C_4 plants to the diets of prehistoric bison as inferred from the stable carbon isotope composition of bone collagen does not directly reflect the expected changes in forage distribution during different climatic episodes. This is probably due to the complicating influence of bison behavioural responses to changing ecological conditions.
4. The dietary response of bison to hot/dry climate conditions does not appear to be consistent through time.

Chapter 4. BEHAVIOURAL RESPONSES TO ECOLOGICAL CHANGE: BISON POPULATIONS OF SOUTHERN SASKATCHEWAN DURING THE HOLOCENE

4.1 Introduction

For nearly a half century, archaeologists have debated the nature of human responses to the Mid-Holocene climate of the Great Plains (Mulloy 1958; Hurt 1966; Reeves 1973; Frison 1975; Forbis 1992; Sheehan 1995). As the single most important animal resource within this region, bison have figured prominently in this discussion. Herd movements and other aspects of bison behaviour would had a significant impact upon the decisions made by contemporary human populations (Roe 1951; Bryan 1991). Nevertheless, our understanding of bison responses to the environments of this period is incomplete (Bamforth 1988; Frison 1991). Models based upon modern and historic populations have proven to be inadequate. The few bison herds which remain in the plains occupy only a fragment of the species' former range and, given their managed and largely restricted nature, offer a poor analog for prehistoric populations (Jones *et al.* 1983; Truett *et al.* 2001). Historic records, while providing a window on the vast populations of the past, are often incomplete and can be difficult to reconcile with other historic accounts and modern field observations (Cannon 2001).

Analyses of archaeological and paleontological remains may be the only way to access previously unavailable information of direct relevance to prehistoric bison populations. Unfortunately, this goal has also proven to be somewhat elusive. By necessity, paleoecological studies involve the analysis of multiple proxies (Vance *et al.* 1995; Sauchyn 1997; Lemmen and Vance 1999). These sources are often resolved to differing temporal scales and can be difficult to associate with one another (Vance *et al.* 1995; Lemmen and Vance 1999). However, stable isotope analyses provide a method through which a variety of data concerning an animal's environment and diet may be discerned from a single dateable fossil (Goh 1991; Koch *et al.* 1994). Isotopic

information derived from bone collagen provides a basis for inferences into trends over the lifetime of adult individuals (Hall 1961). These data may be used to compare the life-ways of animals from different contexts and thereby, identify differences of habitat and diet.

Stable isotope analyses were subsequently performed upon bison remains from six archaeological sites of differing age. The results provided a comparative model of climatic regimes and associated bison diets throughout the Holocene of southern Saskatchewan. An analysis of these data suggests that the foraging behaviour of Mid-Holocene fossil bison may have differed from that of their more recent counterparts. In addition, the reconstructed climate data indicate that the Middle Holocene may have been subject to a degree of temperature variability, despite the persistence of regional aridity throughout the period. These interpretations may facilitate an understanding of the adaptive, distributive and demographic changes that affected bison and human populations throughout the Mid-Holocene on the Northern Plains.

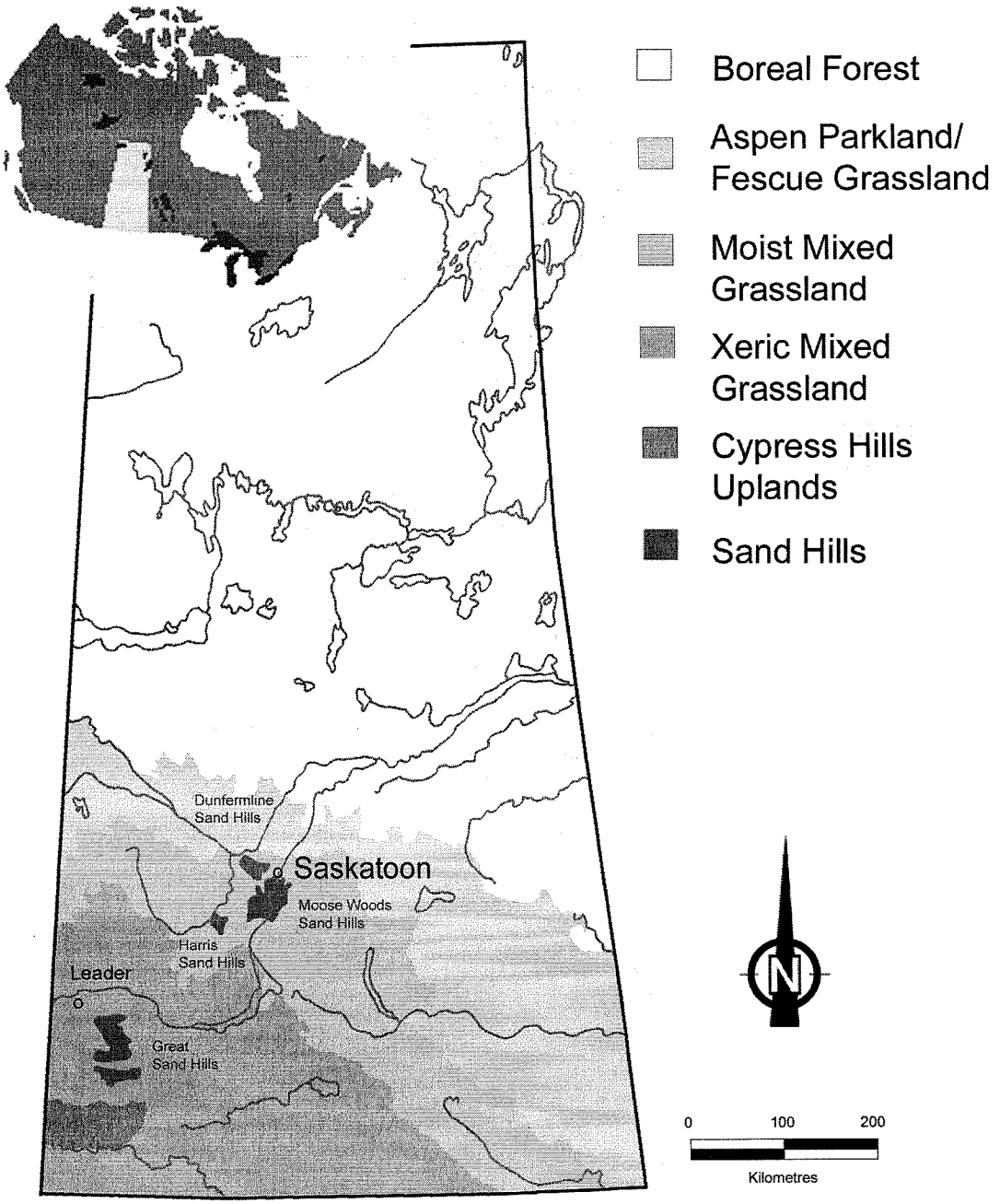
4.2 Modern Ecological Relationships

4.2.1 The Southern Saskatchewan Environment

The Prairie ecozone comprises the southern third of Saskatchewan (Figure 4.1) and covers roughly 240,000 square kilometers from the border with the United States north to the Boreal forests (Acton *et al.* 1998). The region is essentially level with rolling plains and subdued uplands dispersed throughout. The local climate is continental, ranging from semiarid to moderately humid with long, cold winters and short, hot summers. Across this region temperatures tend to decrease with corresponding increases in either altitude or latitude. Precipitation is generally low but levels rise moderately toward the northern and eastern margins. These temperature and precipitation gradients have led to the establishment of climatic zones ranging from cool, semiarid in the southwest to moderately cold, subhumid in the northeast (Acton *et al.* 1998).

The grasslands of southern Saskatchewan are dominated by the fescue and mixed-grass prairies (Morgan 1980; Acton *et al.* 1998). In the deep southwest, the Cypress Hills Uplands constitute an elevated plateau characterized by greater levels of

Figure 4.1 The Ecoregions of Southern Saskatchewan (adapted from Acton *et al.* 1998)



humidity, cooler temperatures and a mixed vegetation comprised of prairie and montane species. The surrounding Xeric Mixed Grasslands are considerably warmer and drier. Vegetation includes a combination of short grasses such as blue grama grass (*B. gracilis*) and a variety of "mid to tall" grasses (Looman 1983a). Northward and eastward, this zone gives way to the Moist Mixed Grasslands dominated by taller grasses, shrubs and sporadic aspen groves. This area is characterized by more moisture, somewhat reducing drought severity. The northern and eastern margins of the grasslands are dominated by the Aspen Parklands which include a mosaic of aspen stands surrounding small wetlands within the rough Fescue Grasslands. Winters in this region tend to be longer and colder, and late summer water deficiencies are much less severe (Coupland 1961; Acton *et al.* 1998).

In a very general way, plant distributions vary regularly across these different ecoregions. On average, the relative proportions of either cool or warm-season species are distinctive for each grassland zone (Coupland 1961). Cool-season plants are those that experience their most vigorous growth and thus their highest nutritional quality, in the spring (May through June) while warm season species are most viable throughout the summer (July through August) (Hanson 1984). These differences in adaptive potential result primarily from the specific type of photosynthesis used by the different plant species (Ehleringer 1991). Most cool-season plants employ C_3 photosynthesis during which atmospheric CO_2 is reduced to a three-carbon compound (Boutton 1991b). The Canadian grasslands are temperate in nature and are thus, dominated by cool-moist season (C_3) species (Boutton 1991b, Tieszen *et al.* 1997a). In contrast, warm-season plants employ a photosynthetic process through which CO_2 is reduced to a four-carbon compound (Boutton 1991b). C_4 species often dominate warm, tropical settings and become progressively rare as latitude and altitude increase (Boutton 1991b, Knapp and Medina 1999).

The availability of moisture limits the overall success of both C_3 and C_4 plants (Buchner 1980; Strahler and Strahler 1992). The dominance of C_4 species in warm climates and their increased competitiveness during the warm-season results from physiological adaptations that optimize the C_4 photosynthetic reaction at higher temperatures and limit it at lower ones (Long 1999). Thus, the most productive climates

for C_4 species in terms of their quality and propagation, would include increased levels of precipitation combined with warm temperatures (Qi and Redmann 1993). Under such optimal conditions, C_4 biota possess a higher overall production potential than do C_3 species (Long 1999). Additionally, the unique physiology of C_4 plants allows them to use available water and nitrogen more efficiently (Knapp and Medina 1999). This higher water-use efficiency enables them to better tolerate drought (Barnes *et al.* 1983). Nevertheless, this physiological advantage does not assist with the overall establishment of C_4 plants during arid periods (Long 1999; Sage *et al.* 1999). Instead, those plants which are established by the onset of drought experience only a limited nutritional loss relative to the large decreases that may be experienced by C_3 biomass (Ozturk *et al.* 1981).

In the temperate Canadian plains, most of the annual cycle is characterized by conditions which are favourable to C_3 growth and establishment. It is for this reason that they dominate overall production and tend to be of higher average nutritional quality (Heckathorn *et al.* 1999). Moist and reasonably cool climates will favour the growth and succession of C_3 species. On a seasonal basis in the Canadian plains, it is only in the late summer that C_4 grasses become competitive. Their preference for warm temperature and ability to tolerate drought allows them both to grow rapidly during the moist spring and to mediate moisture deficits which are common in the late summer (Long 1999). This competitive advantage under water stress is reliant upon the occurrence of warm temperatures and is subsequently lost as climate cools. As a result, environments which are cold and arid are significantly detrimental to the production and distribution of both C_3 and C_4 grasses (Long 1999).

4.2.2 Bison in the Northern Plains Environment

Across their known range, bison can be found to occupy a wide variety of habitats (Jones *et al.* 1983). Although their behaviour is known to vary in response to a range of biotic and abiotic criteria, dietary concerns probably exert the most constant influence (Singer and Norland 1994; Hart 2001). The composition and movements of local bison herds are largely determined by the distribution and quality of vegetation within a region (Vinton *et al.* 1993; Steuter *et al.* 1995; Fortin *et al.* 2002). These forage

characteristics are in turn, controlled by aspects of local and seasonal climate. However, the overall relationship between climate, the characteristics of regional forage, and the behaviour of local bison populations may be quite variable from place to place. As a result, bison herds may exhibit foraging tendencies and preferences that are quite specific to the area that they inhabit (Moodie and Ray 1976; Bamforth 1988). The applicability of any given explanation for bison behaviour may therefore be limited to a particular region.

The temperate Northern Plains are dominated by cool-season (C_3) grasses (Tieszen *et al.* 1997a). While absolute distributions vary somewhat across the ecoregions, C_3 species account for an average about 80% of the available grassland forage in Southern Saskatchewan (Coupland 1961; Tieszen 1994; Tieszen *et al.* 1997b). In addition to their higher distribution, their proclivity for cool temperatures makes them perennially more nutritious than local warm-season grasses (Heckathorn *et al.* 1999). As a result, the diets of local bison will always be dominated by the consumption of C_3 foliage. It is only during the late summer when temperatures rise and moisture deficiencies become frequent that the degrading quality of C_3 species allow for C_4 plants to become a competitive source of food (Ozturk *et al.* 1981). Throughout this period, bison experience rising levels of water and dietary-stress that force them to select for the most nutritious forage species available. Under such conditions, C_4 plants may grow to account for 80% of all species consumed, though only for a very limited period of time (Tieszen 1994; Steuter *et al.* 1995).

Although the diets of Northern Plains bison appear to reflect regional vegetation distributions, they are actually more representative of the overall time which bison devote to the consumption of different forage species. This situation may be limited to peripheral areas of the plains, such as the Canadian prairie, where the relative distributions of warm and cool-season plants are highly unbalanced. In locations such as the Canadian plains, the period of C_4 competitiveness is so restricted, and the abundance of C_3 forage is so great, that changes in plant distribution have a lesser role in the determination of annual consumption patterns among bison. Furthermore, sustained changes in plant distribution usually require a period of sufficient moisture, associated with appropriate temperatures, to allow for the increased establishment of

various plant species (Looman 1983b; Bamforth 1988). In an environment with adequate moisture, most plants reach seasonal levels of nutrition which are sufficient that bison do not experience the amount of dietary stress that is required to motivate significant a change in dietary patterns. In such a context, bison may forage in a reasonably indiscriminate fashion, adjusting their diets only to take advantage of the seasonal differences between C_3 and C_4 vegetation (Hanson 1984).

Among bison populations in southern Saskatchewan, dietary changes are most likely to occur in response to specific episodes of water and dietary-stress. The most important factor determining the extent of these pressures is the regional availability of moisture (Bamforth 1988). Aridity tends to degrade the nutritional quality of growing grasses and can lead to subsequent decreases in density and overall ground coverage (Buchner 1980). Under such conditions, the tolerances of bison for low quality forage may be exceeded. Like most ruminants, they respond to such periods by engaging in a variety of compensatory behaviours (Bamforth 1988). Ambient temperatures play a role in determining the specific nature of such responses.

Seasonal droughts are a pervasive characteristic of the Northern Plains environment (Acton *et al.* 1998). During the late summer, frequent periods of moisture deficit combine with warm temperatures to create a context in which cool-season grasses grow dormant and warm-season species briefly become the most nutritious source of available food. Bison respond to these changes by dramatically increasing their reliance upon C_4 plants, which, by virtue of their drought tolerance and proclivity for warm temperatures, remain active (Steuter *et al.* 1995). It is during this period that bison ingest the majority of C_4 grasses which they consume on an annual basis. In doing so, they are attempting to reduce nutritional stress by maintaining the quality of their diets above minimal tolerances. This tendency is an essential adaptation to the seasonal cycles of the Northern Plains. It distinguishes bison from a variety of other large ungulate herbivores and has allowed them to sustain large populations within a biome that is generally dry (Bamforth 1988).

Many ungulates respond to reductions in forage quality by increasing their overall consumption of vegetation. However, large ruminants are somewhat constrained in their ability to compensate in this fashion. Although efficient, ruminant digestion is a

slow process. There is a rate of consumption beyond which ruminants such as bison, do not gain substantial nutritional benefits (Bamforth 1988). As a result, they must selectively forage for higher quality sustenance during periods of local forage desiccation. In many instances, this requires that they substantially increase their range in the search for better food (Reher 1978; Bamforth 1988). Thus, during periods of nutritional stress, many ruminant populations will disperse across the landscape with small groups or even individuals becoming much more transient. During late summer drought, bison of the Northern Plains are able to avoid this pattern by concentrating on the best source of food which is locally available (Hanson 1984). Their ability to do this, is a direct result of their tolerances for low quality forage.

Nevertheless, there are climatic episodes that bison populations will have some difficulty mediating. In addition to dramatically reducing the nutritional quality of regional forage, particularly severe droughts can result in a loss of overall plant coverage (Heckathorn *et al.* 1999). If, for example, there are insufficient amounts of C_4 forage to sustain resident herds throughout a significant summer drought, they may have to engage other forms of compensatory behaviour; although, the physiology of C_4 plants does render them somewhat resistant to losses of coverage during hot, arid periods (Sage *et al.* 1999). Additionally, regional bison populations should be able to endure some loss of C_4 biomass providing that they are not already over the regional carrying capacity (Reher 1978).

The most detrimental climate, however, is probably one that is both cool and arid. The advantages of warm and cool-season plant species alike are negated in such an environment (Buchner 1980). Cold-arid steppes and temperate desert habitats tend to be characterized by these conditions and are among the least productive biomes in the world (Strahler and Strahler 1992). Under such circumstances, selective consumption would do little to help bison sustain the nutritional value of their diets. As a result, they would have little choice but to begin searching for better foraging patches. In doing so, large populations would probably begin to disperse and the movement patterns of the resulting herds would become less regular and somewhat unpredictable (Bamforth 1988). Survival would be dependant upon an immediate ability to withstand water and dietary-stress and eventually, the discovery of new foraging grounds. In addition, the

duration and severity of the associated climatic episode would play a large role in determining mortality rates and the permanency of resulting population changes (Bamforth 1988).

These interpretations of bison behaviour are based upon modern observations and historical records of bison populations from across the Great Plains. Although imperfect, they provide a conceptual framework from which the responses of modern bison populations to their lifetime environmental context may be predicted (Table 4.1). According to this model, specific changes in local climate are hypothesized to have certain generalized effects on various key aspects of regional vegetation. Specific habitat changes occurring over the lifetimes of local bison should result in a variety of stresses, each of which elicits a distinct response from the affected population. While the relationships between bison diet, climate, and local forage conditions are not direct, bison consumption patterns do relate to the cumulative stresses which result from these criteria. As a result, the average lifetime composition of bison diet should be related to both local climate and the state of regional forage, in a regular and generally predictable fashion.

4.3 Mid-Holocene Influences

4.3.1 Climate Change

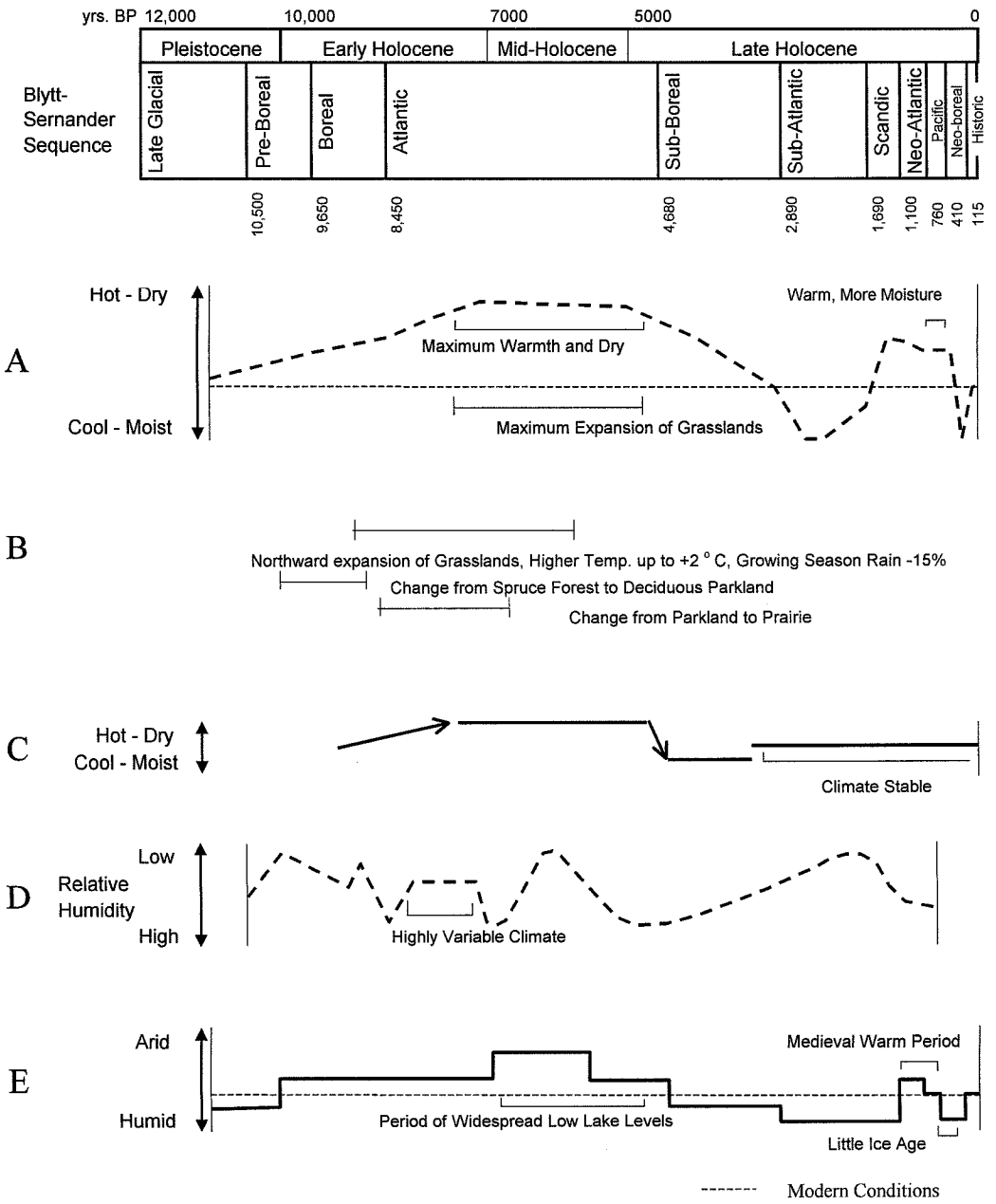
The modern character of the Northern Great Plains has been largely determined by a specific pattern of changing climates that began toward the end of the last glaciation (Figure 4.2) (Dyck 1983). The most important outcome of these events was the gradual establishment of the grasslands. By 10,000 years BP, the area now known as the Canadian plains had begun to change from a region of deciduous forests into a parkland habitat (Yansa and Basinger 1999). Although the climate of this period is poorly understood, this transition is thought to have resulted from the commencement of a warming and drying trend that was to continuously dominate the first half of the Holocene. During this time, the grasslands continued to extend into their present range and by the peak of this trend had expanded to the north and west well beyond their modern boundaries (Vance *et al.* 1995). Using the adopted European Blytt-Sernander

Table 4.1 Predicted Responses of Modern Bison Populations and Regional Vegetation to Lifetime Environmental Trends in the Temperate Canadian Plains

Climate		Regional Forage						Bison Populations	
Relative Temp.	Relative Moisture	Relative Distribution [A]		Relative Nutritional Quality [B]		Relative Coverage [C]		Relative Stress [C,D]	Responses [C]
		C_3	C_4	C_3	C_4	C_3	C_4		
High	High	Decrease (<80%)	Increase (>20%)	Highest	High	Dense	Dense	Low	Limited herd movements and overall range size. Movements regular. High population densities and large herds. Reasonably indiscriminate foraging that is C_3 dominated in fall, winter, spring and C_4 dominated in late summer.
Low	High	Increase (>80%)	Decrease (<20%)	High	Moderate	Moderate	Moderate	Low	Similar to above.
High	Low	Stable (~80%)	Stable (~20%)	Low	Moderate	Sparse	Moderate	Moderate	Depending on duration/severity of climate episode, some transience with movements much less regular. Disproportionate reliance on C_4 grasses.
Low	Low	Stable (~80%)	Stable (~20%)	Low	Lowest	Sparse	Sparse	High	Substantial increases in overall range size and transience. Low population densities and small herds. Attempts made to increase overall consumption. Forage selectivity provides little benefit.

[A] Sage *et al.* 1999 [B] Heckathorn *et al.* 1999 [C] Bamforth 1988 [D] Knapp and Medina 1999

Figure 4.2 Holocene Climate Change in Southern Saskatchewan: A Comparison of Recent Models



A - adapted from Dyck 1983

B - adapted from Vance *et al.* 1995, Vreeken 1999, Yansa and Basinger 1999

C - adapted from Sauchyn and Sauchyn 1991

D - adapted from Shang and Last 1999

E - adapted from Lemmen and Vance 1999

climate sequence (Bryson *et al.* 1987), this period is generally referred to as the Atlantic Climate Episode (~8,500 - 8,700 yrs. BP). The subsequent interval during which Atlantic climates reached their most extreme is often identified as the "Altithermal" or " Mid-Holocene Climatic Optimum" (Walker 1992).

The ecological effects of the Atlantic climate pattern have been widely identified by a variety of paleoenvironmental proxy records from across the Northern Plains. In southern Saskatchewan, sedimentary analyses from a number of lake deposits indicate a widespread occurrence of low lake levels throughout this period (Sauchyn and Sauchyn 1991; Sauchyn 1997; Lemmen and Vance 1999; Birks and Remenda 1999; Richmond and Goldsborough 1999; Vreeken 1999). Other sources including palynological records and plant macrofossils further substantiate this event (Vance *et al.* 1995; Yansa and Basinger 1999). In conjunction, these data have been used to postulate not only pervasive aridity, but also significantly elevated environmental temperatures (Dyck 1983; Lemmen and Vance 1999). It has been suggested that throughout the Atlantic period and particularly during the Altithermal, the northern grasslands became substantially more xeric in nature.

On a seasonal basis, this may have resulted in summertime conditions that included extended periods of hot aridity accompanied by high winds (Vance 1987). Winters may have become comparatively short, although characterized by a higher degree of precipitation relative to modern amounts (Vance 1987). These changes, particularly the reduction of growing season precipitation, would have had detrimental effects on regional forage production. While, almost all vegetation would have experienced some degree of water-stress, cool-season species would have particularly suffered. This would have entailed a loss of nutritional value and a concomitant reduction in the density of regional plant coverage (Reher 1978). Although the Mid-Holocene did include an increase in the total grassland area, these qualitative changes would have probably resulted in a reduction of the regional carrying capacity (Reher 1978). Furthermore, the duration of the Atlantic episode would have almost certainly increased the intensity of this effect (Reher 1978).

4.3.2 Bison Adaptations

Although bison are known to have been present in North America for at least 200,000 years, much of their adaptation to the grassland setting is thought to have occurred only recently (McDonald 1981; Bamforth 1988). During the Early Holocene, contemporary bison populations began rapidly to exploit the abundant source of new forage in the emerging Great Plains. This change in subsistence patterns resulted in pressures that motivated a variety of demographic and morphological refinements. The modern plains bison (*Bison bison*), is thought to derive from a Late Pleistocene progenitor (Wilson 1978; McDonald 1981; Frison 1991). While there is little agreement as to the taxonomy of the various pre-Holocene bison forms, most apparently did not survive the habitat changes associated with the Holocene/Pleistocene transition. Those bison that did survive this period are assumed to have possessed traits of benefit in their new environment.

A variety of Early Holocene bison forms have been proposed, however, there are two commonly described variants, referred to here as *Bison antiquus* and *Bison occidentalis* (Frison 1991; Wilson 1994). While the exact relationships between these species remain undefined, they are commonly differentiated on the basis of their temporal occurrence and specific morphology. *B. antiquus* is the older and typically larger of the two forms and appears to have roots in the Late Pleistocene. *B. occidentalis* first appears around 10,000 years BP and becomes increasingly visible into the Mid-Holocene (Frison 1991). Significantly, by the onset of the Altithermal, the distribution of these two forms appears to have become allopatric, with *B. occidentalis* more common in the Northern Great Plains and the remnant herds of *B. antiquus* to the south of this region (Wilson 1969; Frison 1991). By 5,000 years BP, both *B. antiquus* and *B. occidentalis* are no longer identifiable and the modern *B. bison* appears to replace them within their former range (Wilson 1978).

The rise of *B. bison* throughout the Late Holocene is coincidental with the full establishment of the modern grasslands (McDonald 1981; Bamforth 1988). The physical differences used to differentiate the successive bison taxa of the Holocene are thought to be indicative of associated adaptive pressures. The most visible trend in the evolution of bison, both before and throughout the Holocene, has been a steady decrease in size (Wilson 1978; Frison 1991). Variation toward this end has been noted

among several specific traits. It is apparent from fossil evidence that the average spread of bison horn cores has decreased throughout the Holocene and teeth have generally grown smaller (Guthrie 1970; Wilson 1978; McDonald 1981; Frison 1991). In addition, bones of the distal limb have apparently become more gracile, although there is some disagreement as to whether they have proportionately lengthened or shortened (McDonald 1981; Bamforth 1988; Frison 1991). While there has been debate as to the exact causation of these specific changes, they are usually assumed to be integrated with shifts in dietary and social behaviour (Bamforth 1988).

Climatic changes concurrent with the ending of regional glaciation and the subsequent establishment of the grasslands probably resulted in variation of resource distributions, forage quality and competition among bison (Guthrie 1970, Wilson 1974, McDonald 1981, Bamforth 1988). Those individuals that survived the Holocene/Pleistocene transition probably did so because of an adaptable nature that allowed them to prosper under a variety of conditions. In comparison to the modern prairies, the cooler and somewhat moister Late Pleistocene environment of the Northern Plains probably included several niche habitats supporting a diverse, but limited amount of high nutrition forage (McDonald 1981). These conditions would have been best suited to small populations of bison capable of a diverse pattern of subsistence. Only by engaging in a mix of browsing and grazing, could large-bodied animals efficiently consume the large amounts of forage required to maintain good nutrition and subsequently, prosper in such an environment. It has been proposed that the characteristic morphology of *B. antiquus* is consistent with that of a bison form adapted to a mixed-diet (McDonald 1981; Bamforth 1988). While its robust, elongate form made it an efficient competitive browser, its ruminant biology and smaller size relative to its predecessors, helped it to exploit sporadic grazing patches of the transition period.

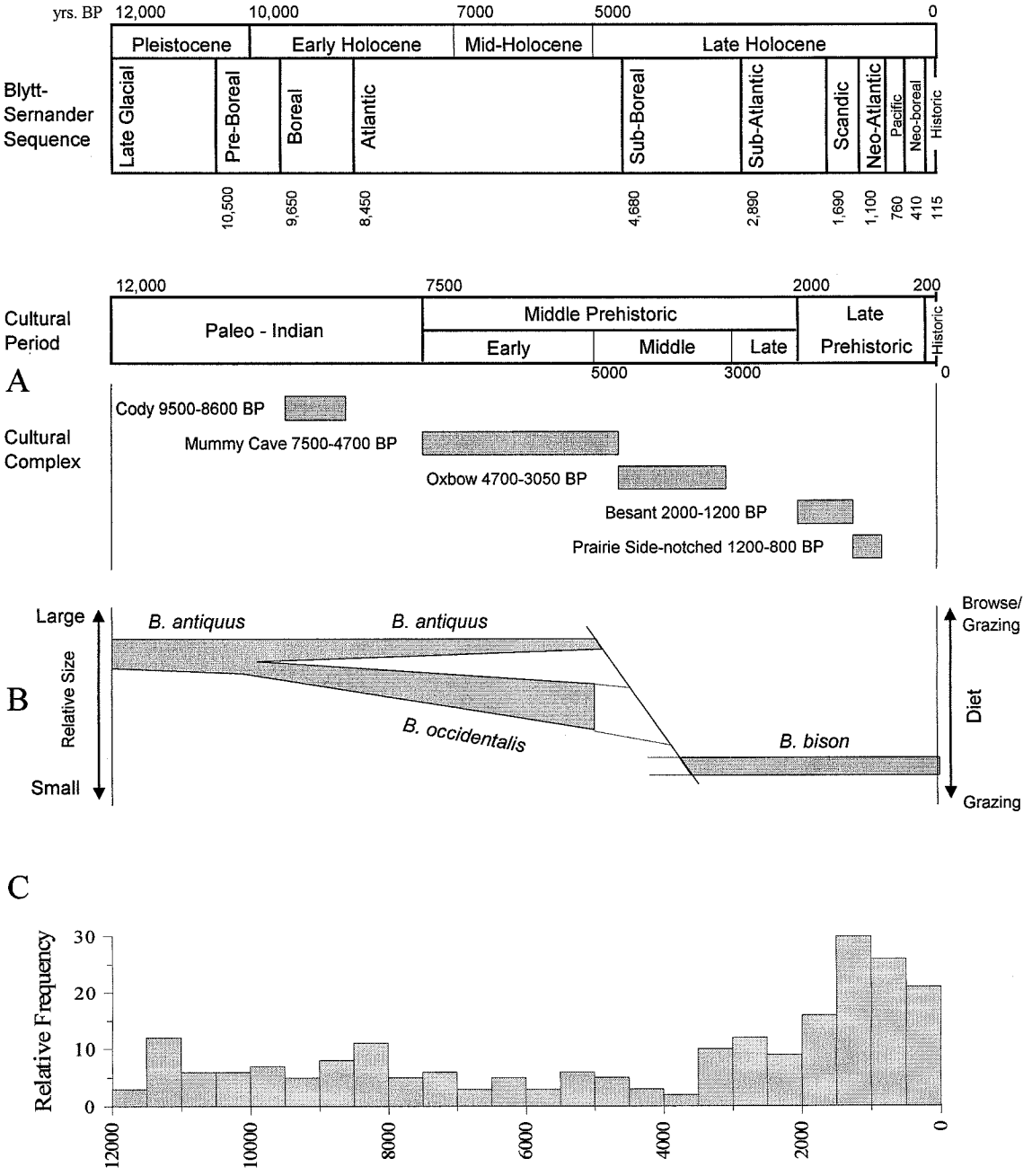
Nevertheless, the continued expansion of the grasslands throughout the Early - Holocene would have increasingly favoured grazing animals that could tolerate a lower overall quality of forage. Many of the subsequent morphological differences exhibited by *B. occidentalis* and eventually *B. bison* are thought to represent a continuous gradient of adaptation towards such a specialization (McDonald 1981; Bamforth 1988). In addition to a generalized size diminution, the occipital region of the bison skull

appears to have reorganized through time, reflecting a decrease in average cranial mass and a generalized downward rotation of the head relative to the vertebral axis (McDonald 1981; Bamforth 1988).

Other changes include an apparent increase in relative orbital protrusion and a lateral re-orientation of the orbits from high on the front of the head (Arthur *et al.* 1975; McDonald 1981). A lower held head quite possibly reflects a shift from eye-level browsing toward low-level grazing; while laterally placed eyes may have facilitated herd coordination and predator monitoring during the consumption of graminoid forage. Concomitant changes in horn core morphology including a decrease in tip-to-tip spread and changes of orientation involving a backwards deflection with increasing curvature are thought to be related to a generalized increase in gregariousness and herd cohesion, lessening the risk of injury during competition among congregating animals (McDonald 1981; Bamforth 1988). Such pressures would have been increasingly brought to bear as bison were forced to concentrate in pockets of refugia during the Mid-Holocene and eventually as large herds began to congregate on the open plains during the late Holocene.

In any event, pressure towards an intensive grazing subsistence would have probably reached a zenith during the Mid-Holocene as the grasslands expanded to their maximum extent under the prevailing climate regime (Reher 1978). The nutritional changes brought by the increasingly xeric conditions may have modified or even increased dietary pressures to a point that challenged the tolerances of contemporary bison taxa. An analytical comparison by Wilson (1978) on specific skeletal measurements from a variety of fossil bison suggests that the rate of Holocene dwarfing may have briefly increased during the Altithermal period. It has been proposed that this trend may have resulted from a corresponding increase in selective pressure for a particularly beneficial phenotype (Figure 4.3). However, the most pervasive and debated evidence for the nature of Mid-Holocene bison adaptations has come from analyses of the archaeological frequency and distribution of bison remains and bison kill sites.

Figure 4.3 Holocene Changes Among Human and Bison Populations



A - Paleocultural Sequence For Saskatchewan; adapted from Linnamae *et al*. 1988

B - Adaptations and the Temporal Relationships Among Bison Taxa of The Holocene; adapted from McDonald 1981

C - Relative Frequency of Bison Remains in Archaeological and Paleontological Contexts From Across the Northern Plains Throughout the Holocene; after McDonald 1981

4.3.3 Population Responses

Since at least the 1950s, researchers studying the prehistory of the Great Plains have periodically noted a conspicuous paucity of identifiable cultural materials from contexts that date to within the Mid-Holocene period. It has been widely suggested that this phenomenon provides evidence of a significant alteration in the settlement patterns of contemporary human populations (Frison 1991). In addition, the subsistence practices of these peoples also seem to have changed. The relative frequency of communal bison kills, as well as the magnitude of each such event, appears to have been lower than at any other time during the Holocene (Frison 1991; Forbis 1992). While a variety of factors are thought to have contributed to these effects, almost all hypotheses acknowledge the significant impact of Mid-Holocene climate changes. The de-stabilizing influence of the increasingly arid and potentially warmer environment are thought to have altered the area and productivity of the grasslands to an extent that generated a systemic response in both bison and human populations (Reher 1978). Regardless, the debate concerning the exact nature of these responses has been considerable.

The earliest attempts to explain the poor archaeological representation of bison and human activity during the Altithermal period, promoted the concept of an almost complete abandonment of the Great Plains (Mulloy 1958). Although this idea became extremely influential, it was subsequently discredited as continued research uncovered evidence of at least sporadic occupation throughout this interval (Frison 1975). More recent hypotheses continue to acknowledge the influence of the Mid-Holocene climatic regime, while suggesting a more variable response by bison herds and the human groups which were dependant upon them (Hurt 1965, Frison 1975; Buchner 1980; Forbis 1992; Walker 1992; Sheehan 1995; Brooks-Lovvorn *et al.* 2001). The notion of "refugia" has figured prominently in this discussion. During the Altithermal, some regions, including river basins, high elevation areas and localities on the periphery of the open plains, may have been subject to a lower degree of overall moisture deficiency. These areas would have provided respite from the harsh environment of the open grasslands and thus, may have continuously sustained small groups of animals (Hurt 1965; Sheehan 1995). While making use of these "refugia", human populations may

simultaneously have had to diversify their subsistence base in response to the smaller area of productive grassland and the limited availability of bison (Hurt 1966).

While most researchers acknowledge the plausibility of the refugia theory, there have been alternate proposals. In an influential paper, Reeves (1973) suggested that the apparent paucity of the Mid-Holocene archaeological record may in fact be a result of inadequate archaeological sampling. This conclusion was based on the assumption that climates of the period did not have as severe an effect upon contemporary bison populations as had been previously speculated. Reeves (1973) maintained that the primary effect of the Altithermal environment would have been an increase in the area of the short-grass plains. Since modern observations and historical records indicate that bison are extremely well-adapted to this habitat, populations of the Mid-Holocene should have been able to tolerate a shift toward a more xeric environment. Thus, a viable but potentially smaller bison population could have continuously inhabited the open plains.

Unfortunately, subsequent research in the years following Reeve's (1973) initial proposals has failed to provide conclusive support for his theories of sampling bias (Sheehan 1995). It is now largely accepted that major changes in the composition of bison and human populations did occur in response to the climates of the Middle Holocene, even if the plains did continue to be sporadically occupied throughout the period (Walker 1992). Nevertheless, there is some evidence to support Reeves (1973) assertions concerning the abilities of modern bison to tolerate the effects of regional drought. The modern foraging behaviours of bison include a variety of strategies which are used to exploit opportunities in the arid environment of the short-grass plains. The preferential consumption of warm-season grasses during the late summer is probably the most important mechanism by which bison cope with changes in forage quality during hot, seasonal droughts (Hanson 1984). Further evidence for the drought tolerances of anatomically modern bison is provided by the apparent success of populations during subsequent xeric periods in the Late Prehistoric (Reeves 1973; Dyck 1983; Huebner 1991). The apparent growth, or at least stability, demonstrated by bison populations during the Scandic climate episode, suggests an inherent ability to tolerate the ecological effects of sustained periods of hot, dry weather (see Figure 4.3).

If there is a difference in the tolerances of Early Holocene bison and their Late Prehistoric counterparts for hot-arid habitats, direct evidence remains elusive. It has been suggested that bison of the Early Holocene may have had fundamental biological differences from their Late Holocene successors (Bamforth 1988; McDonald 1981). Fossil remains from contexts spanning the last 10,000 years, indicate a steady morphological refinement of the genus. Over time, these differences appear great enough to some researchers to suggest chronologically distinct species. It is possible that these forms behaved in fundamentally different ways to similar ecological stimuli. Alternatively, it has been widely speculated that Altithermal climate changes were of a duration and intensity that was otherwise unrivaled during the Holocene period. The resulting effect upon the condition of local forage may have been so severe as to require an unconventional response by resident bison herds (Reher 1978).

Unfortunately, neither of these theories can currently be validated. Both rely upon behavioural assumptions which are difficult to test using current archaeological techniques. While skeletal remains can yield evidence of morphological change they cannot directly characterize new behaviours which an adapting animal may exhibit. Similarly, changes in the frequency and distribution of archaeological remains may indicate changes in social organization and migration, but do not depict the motivations for such a response. For any specific problem involving adaptation in a prehistoric population, the relationship between habitat and behaviour must be identified before resulting changes may be understood. This will require investigative methods that allow for both the elucidation of prehistoric behaviours and their subsequent comparison with associated environmental context.

4.4 Paleoecological Applications of Stable Isotope Analysis

Analyzing the relationship of environmental stimuli to the behaviour of specific prehistoric populations is difficult and requires stringent time controls (Tieszen 1994). Data sources are often widely diverse and difficult to associate. Environmental proxies usually have a large-scale time resolution on the order of centuries or millennia, and studies of prehistoric animal populations are frequently limited to the analysis of skeletal remains which are often incomplete and imperfectly preserved (Reher 1978;

Frison 1991; Lemmen and Vance 1999). Nevertheless, new techniques are allowing for the derivation of detailed and diverse information directly from osteological remains within well-dated contexts.

Using the stable isotope analysis of different elemental isotopes within the bone collagen of prehistoric bison it is possible to reconstruct aspects of diet and climate for individual bison as well as for temporally specific groups (Koch *et al.* 1994). Changes through time in the dietary responses exhibited by bison populations to specific climates may be used to infer differences in behaviour. Stable carbon isotope data from bone collagen can be used to infer animal diet (DeNiro and Epstein 1978). Almost all plants consumed by bison in the Northern Plains employ either C_3 or C_4 photosynthesis (Peden 1976; Looman 1983a; Steuter *et al.* 1995). There is a dichotomous distribution to the carbon isotope composition of plant species using either of these two pathways (Tieszen 1994). These signals become integrated into the tissues of herbivores as plant species are consumed and tissues which are slow to remodel, such as bone, retain a record of dietary inputs over the greater part of an animal's life (Hall 1961). Thus, the relative contribution of these two plant groups to the lifetime diet of an animal may be inferred from the carbon isotope composition of constituent bone collagen using a simple linear interpolation (see Schwarcz *et al.* 1985). These basic dietary determinations are possible because of the direct relationship between the material consumed by an animal and the composition of its bone tissue. However, the use of diet composition to infer habitat climate may be complicated if the animal from which the bone derives exhibits any form of discriminate foraging (Tieszen 1994).

A more direct assessment of habitat climate may be possible through the use of other elemental isotopes, such as nitrogen, which may also be isolated from bone collagen (Ambrose 1993; Koch *et al.* 1994). The stable nitrogen isotope composition of herbivore bone collagen is derived from dietary inputs in a similar fashion to that of carbon (DeNiro and Epstein 1981a). However, the stable nitrogen isotope composition of plant tissues among those species consumed by herbivores such as bison do not vary as a result of physiological differences related to nitrogen incorporation (Peden 1976; DeNiro 1987). Rather, variation of nitrogen isotope values appears to be related to some facet of environmental aridity (Heaton *et al.* 1986; Ambrose 1991). The tissues of both

plants and animals seem to be sensitive to these aridity effects, although the exact mechanism by which this occurs remains unknown (Ambrose 1991; Ambrose 2000). Physiological responses by mammalian herbivores in arid land environments to water and/or protein stress may possibly result in nitrogen (^{15}N) enrichment of body tissues (Ambrose 1991; Hobson *et al.* 1993). In either case, aridity, whether through a direct impact upon plant tissues, or as result of stress-induced biological responses in animals, appears to be responsible for much of the $\delta^{15}N$ variation noted in the tissues of arid-land herbivores (Pate *et al.* 1998).

Temperature is another component of regional climate, and recent work has demonstrated that the stable hydrogen isotope composition of animal tissues reflects environmental temperature regimes (Cormie 1991; Cormie *et al.* 1994c; also see Chapter 2). Hydrogen is introduced into a local ecosystem through regional precipitation and is subsequently dispersed throughout via the hydrologic cycle. Temperature at the site of precipitation largely determines the stable hydrogen isotope composition of local rains and thus all organic and meteoric water reservoirs within a given region come to reflect these values (Gat 1996). These systemic relationships are particularly strong in arid-continental ecosystems where local precipitation is the dominant source of ambient moisture (Dongmann *et al.* 1974; Ehleringer and Dawson 1992). The hydrogen isotope composition of herbivore tissues is subsequently derived from dietary and drinking water sources that reflect the temperature mediated values of regional precipitation (Hobson *et al.* 1999). In addition, tissues that are slow to remodel, such as bone collagen, provide long term aggregate estimates of lifetime hydrogen inputs. As a result, short term or even seasonal variations are averaged.

In combination, δD and $\delta^{15}N$ values isolated from temporally distinct samples of bison bone collagen may be used to construct a qualitative model of climate change for a given locality. Diet differences between these populations, as inferred from $^{13}C/^{12}C$ ratios, may then be interpreted through a comparison to lifetime climate patterns, as opposed to large-scale climatic episodes. As a result, it may be possible to identify changes through time in the basic foraging behaviour of prehistoric bison at a scale which has relevance to the actual lifetime contexts of distinct individuals and populations. Such observations may provide evidence of the choices made by specific

individuals and herds under specific biological constraints, in the context of the immediate local environment. Such data are needed if archaeologists and paleoecologists are to gain meaningful insight into why these choices were made.

4.5 The Behavioural Responses of Bison to Climate Change

To examine isotopic variation in the tissues of bison through time, bone samples were obtained from six distinct archaeological contexts within southern Saskatchewan and stable carbon, nitrogen and hydrogen isotope analyses were performed (Figure 4.4; Table 4.2). These sample groups ranged in age from approximately 10,300 years BP to 1,050 years BP. Each specimen was prepared according to the method described by Sealy (1986), combusted at 850°C in Vycor tubes, and measured on a Micromass Optima dual-inlet isotope ratio mass spectrometer (see Chapter 3). Corrections for hydrogen exchange were made following the procedure described by Wassenaar and Hobson (2000). All stable isotope ratios are reported using delta (δ) notation in parts per thousand (‰) deviation from a known standard: PeeDee Belemnite (PDB) for carbon, Atmospheric Nitrogen Gas (AIR) for nitrogen, and Vienna Standard Mean Ocean Water (VSMOW) for hydrogen). The approximate percentage of C_4 plants (C_4 %) in the diets of individual bison were calculated from $\delta^{13}C$ values using the following equation (Schwarcz *et al.* 1985):

$$C_4\,\% = \frac{(\delta^{13}C_{\text{measured}} - \delta_3 - \Delta_{dc})}{\delta_4 - \delta_3} \times 100\%$$

where $\delta^{13}C_{\text{measured}}$ is the measured carbon isotope composition of an animal's bone collagen, δ_3 and δ_4 represent the average $\delta^{13}C$ values from within the range exhibited by most C_3 plants (-26.5‰) and most C_4 (-12.5‰) plants respectively, and Δ_{dc} represents the average trophic discrimination (5‰) between an animal's diet and its bone collagen (Tieszen 1991).

The resulting δD and $\delta^{15}N$ measurements indicate that climates of the southern Saskatchewan plains have varied significantly over the approximate 10,000 year span examined in this study (Figure 4.5). While it is not yet possible to quantify temperature

Figure 4.4 Map of the Southern Saskatchewan Showing the Archaeological Sites Used For This Study

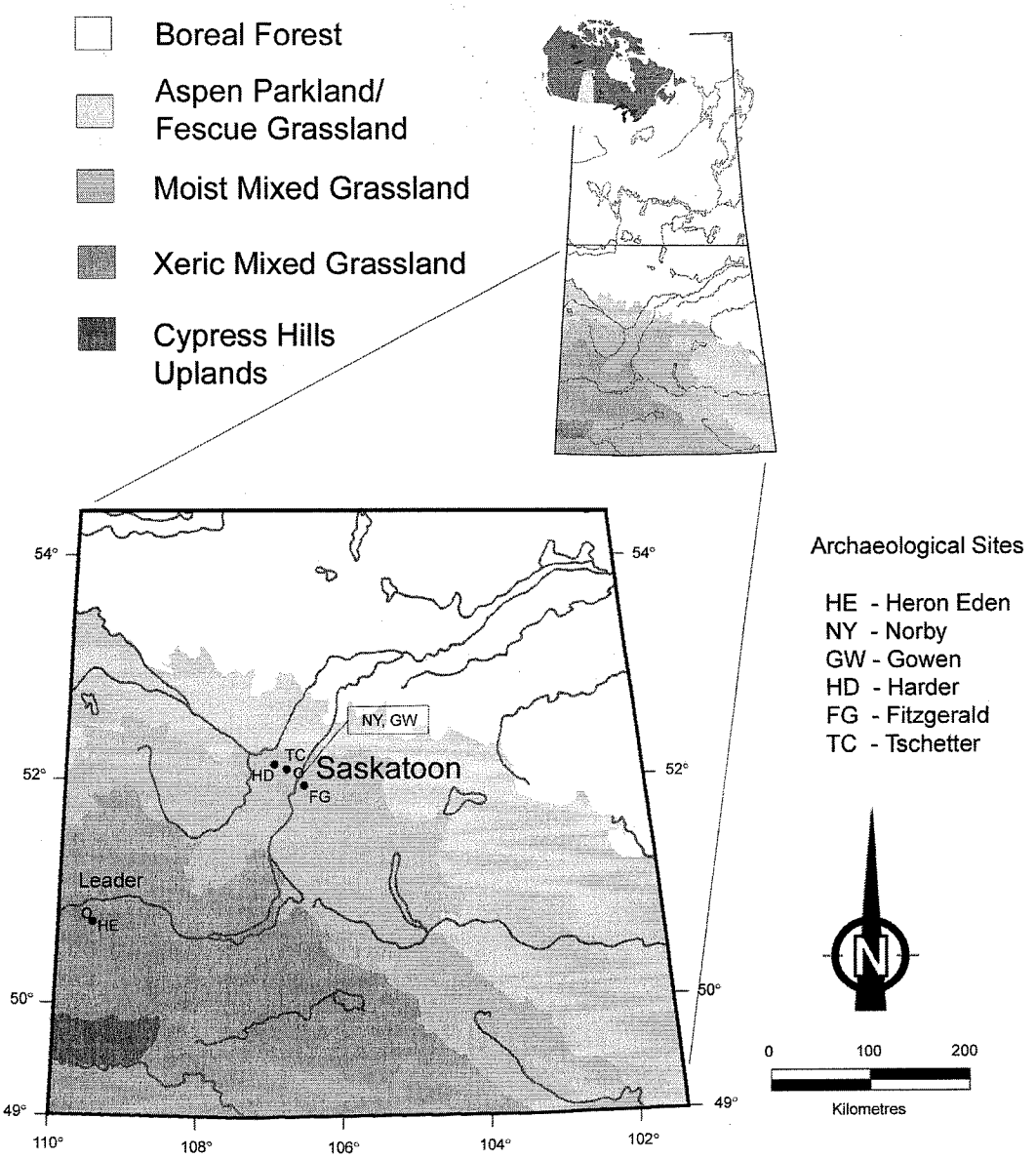
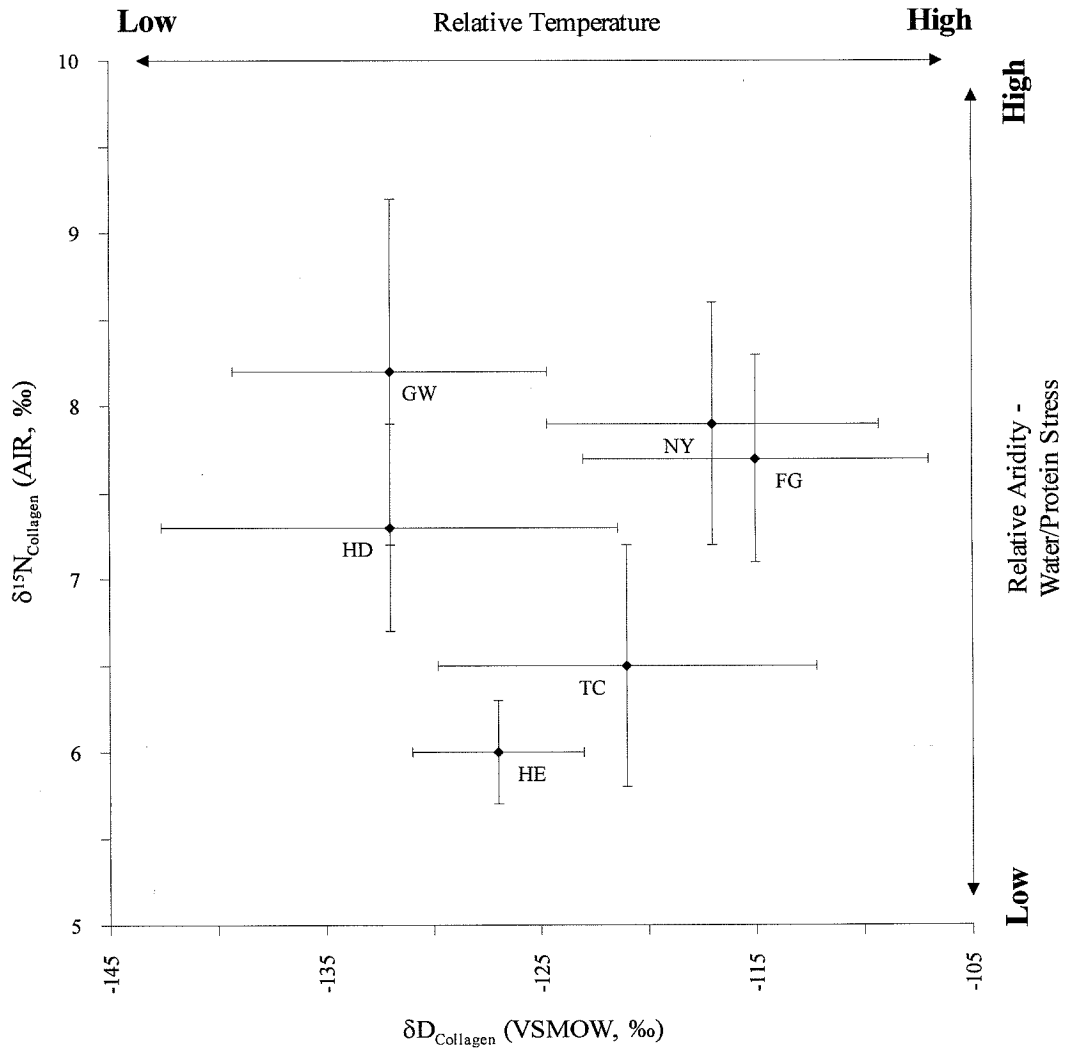


Table 4.2 Mean Bison Bone Collagen Stable Isotope Ratios

Site	Borden Designation	Cultural Association*	Species**	yr. BP (Lab #)	δD (‰)	$\delta^{15}N$ (‰)	$\delta^{13}C$ (‰) (% C_4)‡
Heron Eden (HE) (n = 7)	EeOi - 11	Cody	*B. antiquus*	10,352 ± 134 (NZA 15745)	-127 ± 4.0	6.0 ± 0.3	-19.7 ± 0.4 (13.2 ± 2.6)
Norby (NY) (n=7)	FaNq - 56	Mummy Cave	*B. occidentalis*	7,846 ± 103 (NZA 15747)	-117 ± 7.7	7.9 ± 0.7	-19.0 ± 0.5 (17.8 ± 3.3)
Gowen (GW) (n=6)	FaNq - 32	Mummy Cave	*Bison sp.*	6,653 ± 143 (NZA 15746)	-132 ± 7.3	8.2 ± 1.0	-19.2 ± 0.6 (16.4 ± 4.0)
Harder (HD) (n=8)	FbNs - 1	Oxbow	*B. bison*	4,823 ± 33 (NZA 15776)	-132 ± 10.6	7.3 ± 0.6	-18.8 ± 1.0 (19.2 ± 6.9)
Fitzgerald (FG) (n=10)	ElNp - 8	Besant	*B. bison*	1,442 ± 101 (NZA 15750)	-115 ± 8.0	7.7 ± 0.6	-17.8 ± 1.0 (26.5 ± 7.3)
Tschetter (TC) (n=9)	FbNr - 1	Old Woman's	*B. bison*	1,035 ± 40†	-121 ± 8.8	6.5 ± 0.7	-18.9 ± 0.9 (18.7 ± 6.3)

yr. BP, "calibrated radiocarbon years before present", 2 sigma range, calibrated using INTCAL98_14C, based on Stuiver *et al.* 1998
Lab #, Rafter Radiocarbon Laboratory sample number
* Cultural associations are based upon projectile point associations (see Corbeil 1995; Zurburg 1991; Walker 1992; Dyck 1977; Hjermstad 1996; Linnamae 1988)
** Species determinations based upon previous studies (see Corbeil 1995; Zurburg 1991; Walker 1992; Dyck 1977; Hjermstad 1996; Linnamae 1988)
B., "*Bison*"
Bison sp., Indeterminate species
† The Tschetter date is an average of radiocarbon dates determined prior to this study (see Linnamae 1988)
‡ % C_4 is the percentage of C_4 plants in the average diet of bison calculated from mean $\delta^{13}C$ values

Figure 4.5 Climatic Differences Among the Sampled Time Periods in Southern Saskatchewan as Indicated by δD and $\delta^{15}N$ Values in the Bone Collagen of Prehistoric Bison



*Each Period is Represented By the Calculated Mean of the Sample Population
Error Bars Represent One Standard Deviation

and precipitation changes as indicated by stable hydrogen and stable nitrogen isotope ratios of bone collagen, these values do serve to indicate the basic nature of such environmental shifts (Cormie *et al.* 1994c; Hobson *et al.* 1999). For the most part, the reconstructed climatic patterns for each time period agree with previously proposed scenarios of regional climate change that have been developed from other paleoenvironmental proxies (Dyck 1983; Lemmen and Vance 1999; see Figure 4.2). The only substantial difference is evident from the Gowen site time period. While $\delta^{15}N$ measurements from the Gowen site bison population suggest the occurrence of a comparatively dry climatic episode, corresponding δD values suggest a relatively moderate or even cool temperature regime. While the identification of an arid period during the Mid-Holocene is not unexpected, the suggestion of lower accompanying temperatures is somewhat surprising.

For the most part, the paleoclimate proxies that have been employed in the estimation of Holocene environments from Southern Saskatchewan do not provide a direct characterization of associated climatic temperatures (Vance *et al.* 1995; Sauchyn and Beaudoin 1998; Lemmen and Vance 1999). In many instances, aridity proxies are used simply to infer the occurrence of probable temperature patterns. Hot periods are assumed to correlate with high aridity environments while moderate temperatures accompany moister episodes. The results presented here for the Gowen sites, illustrate the potential problems of employing such an approach. Although arid habitats are generally limiting for the success of plants and animals, those that become inordinately cool for an extended period may produce a high degree of systemic biological stress (Strahler and Strahler 1992; Long 1999).

In the Northern Plains, many plants and animals possess adaptations for coping with seasonally specific drought (Bamforth 1988; Heckathorn *et al.* 1999). Bison are able to tolerate the deleterious effects of summer moisture deficits on regional forage by intensifying their consumption of C_4 grasses, the most nutritional food source available under such conditions. However, the drought-resistant physiology of warm-season C_4 species is only functional during periods with high average temperatures (Long 1999). Alternatively, the perennial cool-season grasses which dominate the region and are the most attractive food source throughout the fall, winter and spring; can only survive late

summer droughts by becoming dormant (Lemmen and Vance 1999). Their success throughout the remainder of year is reliant upon adequate moisture (Sage *et al.* 1999). Thus, the nutritional quality of both C_3 and C_4 plant species is expected to degrade in years that are unusually dry and cool (Long 1999). Given that these plant types together account for almost all regional forage, bison herds of the Northern Plains would be expected to similarly suffer during such episodes.

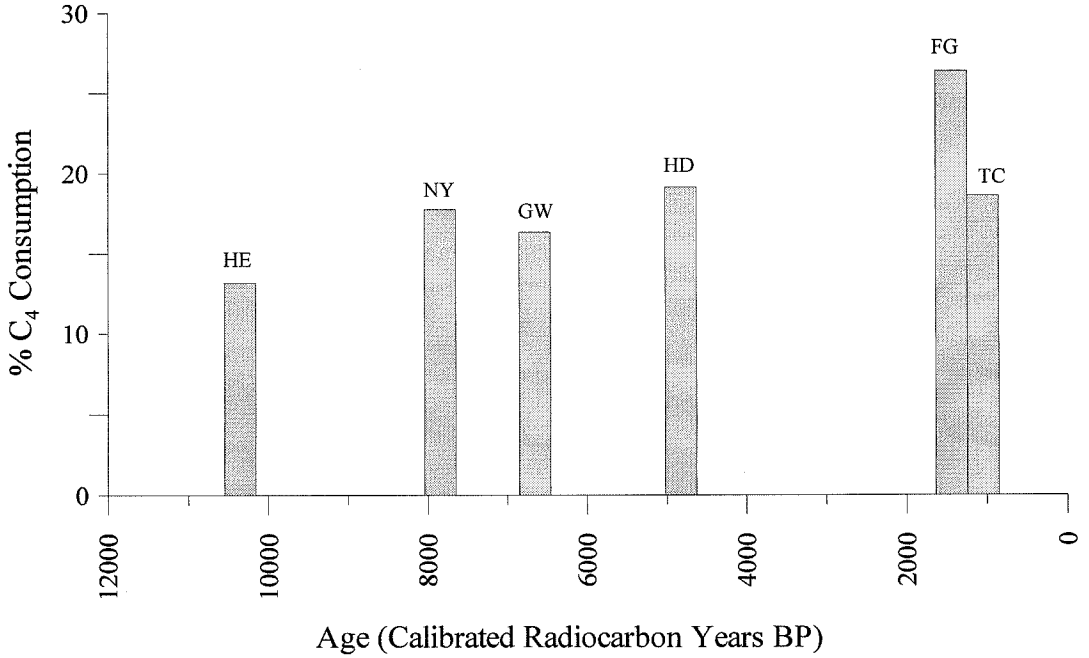
Paleobotanical remains isolated from soil samples taken during the Gowen site excavations indicate a paucity of locally abundant chenopodium, a circumstance potentially indicating a sparseness of vegetation during the prehistoric occupation of the area (Walker 1992). In addition, the highly fragmented nature of the recovered faunal materials has been used to suggest the occurrence of highly stressful climate requiring maximum utilization of all available resources (Walker 1988, 1992; Forbis 1992). These indicators may be particularly significant given subsequent isotopic data which suggests that contemporary climates may have been particularly cool in addition to arid. The severe nature of such a habitat provides a consistent explanation for these phenomena. The Gowen site data thus illustrate the importance of developing proxies that may be used to independently distinguish the temperature and moisture characteristics of past environments. An accurate interpretation of the prehistoric foraging ecology of bison populations requires an understanding of contemporary forage conditions. Since the responses of regional vegetation vary substantially according to climatic context, it is essential that paleoecological models be based upon the most detailed climatic data available.

When such conditions are met, it may be possible to identify and even to explain apparent differences in behaviour through time. Dietary information derived from stable carbon isotope values in the bone collagen of prehistoric bison, may be compared to these environmental data to examine foraging differences between prehistoric bison populations. Since climate is known to have predictable effects upon regional vegetation, bison groups that exhibit distinct dietary patterns and which derive from different time periods possessing similar climates, are almost certainly employing different foraging strategies.

For the most part, the average $\delta^{13}C$ results from the bison populations examined in this study are reasonably stable and appear generally to reflect modern forage distributions within southern Saskatchewan (Figure 4.6). However, the δD and $\delta^{15}N$ results indicate substantial climatic differences among the various time periods. Since forage distributions should change in response to climate, the stability of bison diets through time must be a result of behavioural adjustments by the different bison groups. It therefore appears, that under most climatic regimes, bison engage in behaviours to mediate the impact that climate driven changes in regional forage would otherwise have upon their overall diets (Hanson 1984). Under specific conditions, this mediation of diet is quite predictable from modern observations. During periods with adequate moisture, regardless of ambient temperature, there is a wide variety of nutritionally adequate vegetation available to bison (Sage *et al.* 1999). While temperature differences may induce changes in plant distribution, C_3 species are nutritionally superior throughout most of the year (Hanson 1984; Steuter *et al.* 1995). As a result, bison would experience little pressure to alter their seasonal patterns of consumption irrespective of distributive changes in regional biomass.

During moisture deficient periods, however, bison must respond to varying levels of nutritional stress resulting from climatic interference with vegetation growth. When associated with cool ambient temperatures, these episodes result in a substantial reduction in ground coverage and nutritional value across most forage species (Bamforth 1988; Long 1999). In the face of such pandemic change bison would derive little benefit from altering their reliance upon particular plant species. Instead, they would be better served by foraging indiscriminately in an attempt to ingest as much protein as possible while in pursuit of better pastures (Bamforth 1988). Such a response may well account for the dietary pattern of the Gowen bison. Despite δD and $\delta^{15}N$ values which indicate a cooler and drier environment, $\delta^{13}C$ measurements from the Gowen population do not indicate much variation in terms of the relative consumption of C_3 and C_4 plants. In light of archaeological and climatic evidence indicating a stressful habitat, it seems reasonable to conclude that the Gowen period bison employed means other than dietary selectivity to cope with their environment.

Figure 4.6 Changes in the Relative Consumption of C_4 Plants Among Different Bison Populations Foraging in Southern Saskatchewan During the Holocene



C_4 Percentages are Calculated (see Schwarcz *et al.* 1985) From Mean $\delta^{13}C$ (see Table 4.2) For Each Sampled Population

Although dispersion is a viable response used by a variety of large ungulate species, it is not necessarily the best option available to bison during all instances of drought (Bamforth 1988; Heckathorn *et al.* 1999). On a seasonal basis in the Northern Plains, bison mediate the nutritional stresses of the arid, late summer by intensifying their use of warm-season C_4 grasses (Hanson 1984; Vinton *et al.* 1993; Tieszen 1994; Steuter *et al.* 1995). The physiology of these plant species enables the efficient use of water, thereby lessening the adverse effects of aridity (Sage *et al.* 1999). However, this mechanism is only functional at high relative temperatures and as a result, bison will only benefit from an increase in C_4 consumption during droughts which are associated with warm conditions. Consequently, climatic shifts towards a hotter and drier habitat should cause bison to intensify their overall usage of C_4 species. Since the populations of the Northern Plains only ingest substantial amounts of C_4 grass during a very limited period in the late summer, even a modest increase in the duration of seasonal droughts should result in identifiably greater levels of annual consumption.

The stable carbon isotope analysis of collagen from bison bone samples in the Fitzgerald site archaeological assemblage revealed a higher relative consumption of C_4 grasses than was evident at any other time period. Since both the availability and attractiveness of forage is controlled by environmental criteria, it is highly probable that this pattern reflects at least some influence of the contemporary climate. The Fitzgerald occupation dates to within the Scandic Climate Episode, an interval generally believed to have been characterized by higher temperatures and a low relative moisture availability (Dyck 1983; Lemmen and Vance 1999). The comparatively positive δD and $\delta^{15}N$ values from the Fitzgerald bone substantiates the occurrence of such an environment during the specific period in which these bison lived. Since a shift towards hot, dry conditions should not result in a significant increase in the distribution of C_4 grasses, a higher relative consumption of these species must represent a selective preference on the part of the Fitzgerald bison. In fact, such a response is quite predictable based upon observation of the seasonal patterns which are exhibited by modern bison in the Northern Plains.

However, the results of this study do not demonstrate conclusively that bison react to high temperature periods of aridity in a constant fashion. Although the stable

hydrogen and stable nitrogen isotope composition of bone collagen samples from the Norby site bison assemblage indicate a climate of almost identical character to that of the Fitzgerald period, the $\delta^{13}C$ results do not indicate a similar dietary response. The reasons for this discrepancy are not immediately clear, however, it is certainly not the first piece of evidence to suggest a difference between bison populations of the Early Holocene and their Late Holocene counterparts. The physiological and demographic differences which distinguish bison from these two periods are often thought to reflect the influence of environmental stresses which necessitated biological and social refinements (McDonald 1981; Bamforth 1988). Certain behaviours may well have evolved in conjunction with these other characteristics as a response to the expansion of the grasslands and the increasing need to exploit the seasonal cycles of forage production within such an environment (Reher 1978; McDonald 1981).

The apparent success of bison during analogous climatic periods in the latter Holocene (i.e. the Scandic), does seem to lend support to the suggestion that the severe stresses endured by populations throughout the Altithermal resulted from some type of behavioural, social, or biological incapacity which was subsequently corrected through adaptation. Interestingly, in his analysis of the Fitzgerald site bison assemblage, Hjermstad (1996) reports the presence of an unusual number of older individuals. Such an age structure is unusual for a catastrophic kill event and suggests a reasonably low rate of attrition (Hjermstad 1996). Hjermstad (1996) interprets this to be an indication of an exceptionally healthy population and subsequently proposes that this success was probably encouraged by favourable environmental conditions. Although the data presented in this project contradict this interpretation, depicting the local climate as having been somewhat stressful, the age structure of the population does seem to support the conclusion that the Fitzgerald bison were reasonably fit. It is possible that this success is further evidence of the enhanced suitability of Late Holocene bison for the drought cycles of the short-grass plains.

However, it is also plausible that the unique and severe nature of the Altithermal climate may have precluded the normal expression of certain behaviours by contemporary bison populations. It has been widely speculated that the hot and particularly arid conditions which defined the Altithermal period were of a duration and

magnitude unrivaled in the Late Prehistoric period (Reher 1978; Dyck 1983; Lemmen and Vance 1999). These conditions are thought to have had a nearly catastrophic effect on local vegetation, surpassing even the tolerances of the most drought-resistant of warm-season grasses (Reher 1978). As a result, bison would have been subject to untenable stresses similar to those of a cool and arid habitat. Selective consumption would provide little benefit and many individuals would be unable to survive. Those that did would have had to move abroad in the search for better pastures. Dependant human populations would have eventually followed them.

While the results of this study do not point to a specific cause for the dietary discrepancy of the Norby and Fitzgerald bison, they do lend support to the attribution of a unique severity to the climates of the Mid-Holocene. The changes among subsequent bison populations throughout the remainder of the epoch not only included long-term social and biological reorganization, but apparently also necessitated a difference in the immediate response to localized forage conditions. Regardless of the exact causation, this appears to represent the first direct evidence to suggest that the changes in environment associated with the Altithermal period were beyond the tolerances of contemporary bison populations.

4.6 Conclusions

The effects of Holocene climate change are thought to have been substantial for both human and bison populations. Just as bison were subject to stresses resulting from changing foraging conditions and moisture availability, human populations would have been similarly affected by the same alterations and their subsequent impact upon bison herds. These episodes are visible in the archaeological record as changes in both the nature and frequency of paleontological and archaeological sites. Nevertheless, such information provides only a vague characterization of the forces which acted to create these demographic modifications. As a result, interpretive explanations of these phenomena have been influential but also debatable. Although many archaeologists postulate that the arid Middle Holocene created an environment that was largely inhospitable to bison, others point out that modern herds appear somewhat resilient to the frequent droughts of the short-grass plains. These inconsistencies are difficult to

resolve using the available information. While skeletal evidence suggests that bison physiology has continued to evolve throughout the Holocene, behaviour can only be speculated from such observations. Climate and biology provide only a potential motivation for behaviour and cannot be considered proof of it. Similarly, the distribution and quantity of archaeological sites and skeletal remains results in part from behaviour, but does not necessarily help to characterize its specific nature.

Nevertheless, the findings of this study do provide evidence of a specific change in behaviour. Bison from an Early Middle Holocene age site in southern Saskatchewan do not to demonstrate the same selective foraging tendencies evident amongst a regionally equivalent population from the Late Holocene. Within the context of the arid Altithermal, the failure to increase average consumption of warm-season grasses would have been limiting to overall fitness. While it is impossible to certify that this discrepancy represents an adaptive difference between these populations, the morphological dissimilarities between the two do suggest that such a divergence be expected. The elucidation of a significant behavioural change may be an important discovery for the development of an appropriate model of bison adaptation to the successive climates of the Holocene. Together causative climate, physiological limitation and behavioural incapacity may help to explain the substantial nature of the physiological and demographic changes to which bison were subject during the Holocene.

In addition, this project uncovered evidence of at least one period of unexpectedly lower temperature during the arid Altithermal. Cooler and drier habitats can be inherently limiting to regional vegetation. If such episodes were even a sporadic occurrence during the Mid-Holocene, they could have served to increase dramatically the stresses experienced by both bison and human populations. The discovery that Early Holocene bison may have failed during moisture stress to be adequately discriminate in forage selection, while at the same time being subjected to sporadic periods of unusually temperate drought, provides strong indication that populations faced unprecedented pressures during the Altithermal. It is therefore not surprising that population attrition rates appear to be have grown higher at this time and that herds were forced into an unprecedented migration towards the peripheries of the plains and

other areas of refuge. As a result of these phenomena, human populations would have equally struggled and had little choice but to modify their own subsistence practices by increasing conservation, diversifying their resource base, and relocating in response to the movements of local bison herds.

Chapter 5. SUMMARY AND CONCLUSIONS

Throughout the prehistory of the Great Plains bison were of such importance to the organization of human communities that a detailed understanding of the paleoecology of this species is essential to the study of contemporary human culture. Although archaeological investigations are primarily concerned with aspects of the human condition, there are instances in which supplementary studies of specific animal populations can considerably enhance the understanding of associated human groups (Reitz and Wing 1999). Faunal analyses are typically used to examine the turnover and succession of species, and to reconstruct paleoenvironmental and zoogeograhic history (Lyman 1994). However, in situations where the natural behavioural patterns of these animals significantly influence choices amongst human populations, archaeological interpretive methods may also be adapted to develop models of social or subsistence behaviour within non-human groups.

The inference of behaviour from the static archaeological record is the chief pursuit of "mid-level" archaeological theory (Trigger 1989; Willey and Sabloff 1993). This approach involves the use of analogy to develop models from which interpretations can be made. In terms of bison paleoecology, observations of modern herds and analyses of historic records have provided many of the criteria used to identify social and subsistence behaviours in prehistoric bison populations. Unfortunately, this method has had limited success. The restricted and managed nature of modern herds along with the inadequacies of historical sources makes their comparison to prehistoric contexts difficult. In addition, bison are known to have undergone a continuous morphological, and presumably also physiological evolution throughout the Holocene in response to changing environmental and social criteria. These changing parameters must be taken into account if one is to adequately reconstruct contemporary behaviours. However, different paleoenvironmental proxies can be problematic to associate both temporally and geographically. As well, they are seldom resolved to time scales compatible with

the lifetimes of biological organisms. Therefore, archaeologists must establish reliable new measures of the absolute or relative changes that affect these populations and their habitats, in conjunction with a specifically precise chronology to specify the temporal relationships between these changes (Trigger 1989:21).

Stable isotope analyses of fossil remains have been increasingly used to examine aspects of paleodiet and paleoclimate. While these studies have most frequently involved human populations, analyses of archaeological animal bone have been promoted as a valuable method from which to derive ecological context. Research on bison remains has largely involved the reconstruction of diet using stable carbon isotope values in either bone collagen or apatite. However, the results of these investigations have been difficult to interpret. Without the proper context, it is impossible to determine if dietary patterns result primarily from the influence of an animal's behaviour or habitat (Tieszen 1994, Jahren *et al.* 1998)

The objectives of this thesis were three-fold. First, to evaluate the utility of stable hydrogen isotopes in bison bone collagen as an indicator of lifetime habitat temperature. Second, to construct temporally specific ecological models of foraging behaviour through which the reconstructed dietary preferences of bison may be observed relative to reconstructed climatic context. Third, to identify and explain Holocene patterns of bison foraging behaviour among prehistoric herds of the Southern Saskatchewan plains and discuss the implications of these data for archaeological investigations involving contemporary human populations.

Although D/H measurements from animal bone collagen have a demonstrated potential for use as a paleo-temperature proxy, there have been no studies specifically focused upon the use of fossil bison bone as an analytical substrate. A preliminary analysis was conducted using modern specimens to assess the environmental sensitivity of D/H ratios in bison bone collagen and to refine further the technique for specific applications involving archaeological bison bone. The percentage of exchangeable hydrogen in bison bone collagen was experimentally determined using samples obtained from animals born and raised in Southern Saskatchewan. This proportion was found to be approximately 20%, very close to other published estimates for animal bone

collagen. All hydrogen isotope ratios subsequently determined from archaeological samples over the course of this study were corrected using this exchange factor.

Stable hydrogen isotope measurements were made on samples taken from two geographically distinct herds. The δD composition of non-exchangeable hydrogen from modern bison bone collagen was found to demonstrate significant differences relative to location and latitude in continental North America. The isotopic spacing between individuals from southern Saskatchewan and those from an Oklahoma population averaged about 70‰. This separation appears to mirror the continental δD distribution of growing season rain. At both locals, δD values in the tissues of resident bison appear to be consistently offset from the D/H composition of growing season rain by an average of about -35‰. Although dietary samples were collected along with the bone specimens, diet-tissue discrimination could not be quantified as a result of the indeterminate influence of drinking water δD.

Nevertheless, the regional accordance of compositional patterns in both rain and bison tissue, suggests that the stable hydrogen isotope composition of bone collagen continues to reflect the same environmental influences that initially determine the δD value of local growing season precipitation. Thus, variations in the hydrogen isotope composition of bone collagen from distinct populations should provide a reasonable qualitative indicator of differences in habitat temperature, whether as a result of geographic separation or regional climate change. To illustrate this, δD measurements were taken from fossil bone collagen samples representing eight temporally distinct bison populations from archaeological contexts in southern Saskatchewan. The resulting data provided clear indication of a series of significant shifts in the hydrogen isotope composition of bison tissues throughout the Holocene in this region. In most cases, these variations corresponded quite well to the pattern of thermal change predicted by a variety of other environmental proxies.

In at least one instance, however, an important deviation from the expected pattern was noted. Although the Gowen site bison assemblage was derived from a population that lived during the Altithermal interval, the stable hydrogen isotope profile of this group suggested a habitat with a more moderate temperature regime than would otherwise be expected. Two other populations were also dated to this period and yielded

measurements that were more consistent with high environmental temperatures. Although difficult to interpret, this discrepancy may be an artifact of the limited temporal scale represented by the D/H composition of bone collagen. Such measurements provide indication of a very limited period no greater than the lifetime of the source animal. As a result, these data may only represent short-term climate changes and not the larger overall climatic episode. Alternatively, this circumstance may reflect the degree to which stable hydrogen isotope ratios act as a direct gauge of temperature related phenomena. Most conventional climate proxies are substantially influenced by aspects of regional aridity and do not allow for independent assessments of contextual temperature or moisture levels. Therefore, the D/H profile of the Gowen site bison population may differ from expectations derived by other proxies because it is reasonably independent of aridity effects. In any event, this result indicates a degree of temperature variability that is not usually associated with the Middle Holocene.

Subsequent to the analysis of δD values, stable carbon and stable nitrogen isotope measurements were also taken from each of the 52 specimens of fossil bison bone collagen. These data allowed for the interpretation of other aspects of bison ecology; information which could then be contrasted with the temperature data derived from the D/H measurements. Although $\delta^{15}N$ variation among the tissues of individuals from a given species is difficult to interpret, prevailing theories suggest that moisture levels in the lifetime habitat are an important determining factor. This may be particularly true for animals such as bison that consume a reasonably homogenous, non-leguminous diet. Thus, $\delta^{15}N$ measurements may be used to infer differences among lifetime precipitation regimes, or at the very least changes in water and/or dietary stress levels as determined by regional moisture availability. On the other hand, stable carbon isotope values in bone collagen are determined largely by an animal's diet and therefore do not have a direct connection to local climate. Nevertheless, the tissues of herbivorous animals are indirectly influenced by environmental factors which control the availability of local forage. As a result, it is ultimately a combination of environmental and behavioural variables that determine the $\delta^{13}C$ composition of animal bone collagen.

Much like the hydrogen isotope results, the $\delta^{15}N$ and $\delta^{13}C$ profiles from the various prehistoric groups indicate that the stable carbon and stable nitrogen isotope

composition of local bison tissues has varied to some degree over the course of the Holocene in Southern Saskatchewan. Nevertheless, the chronological changes among each isotopic fraction do not appear to be strongly correlated. This finding supports the idea that stable hydrogen, nitrogen and carbon isotope values in bison bone collagen are independently dictated by disparate ecological phenomena. δD and $\delta^{15}N$ values almost certainly reflect climatic context, being respectively influenced by temperature and moisture availability. The observable pattern of change in the nitrogen isotope composition of bison bone collagen, while distinct from that of hydrogen, can nevertheless be reconciled with the general model of aridity variation that is thought to have occurred during the last 10,000 years in southern Saskatchewan. However, the observable Holocene changes in the stable carbon isotope composition of bison bone collagen do not bear strong correlation with either δD or $\delta^{15}N$ patterns and as such, do not appear to reflect aspects of habitat climate directly.

The degree to which the foraging behaviour of bison dictates the carbon isotope composition of their tissues, is probably variable across the North American landscape. Although several studies have found that the stable carbon isotope composition of bison tissues tend to reflect a diet in which the relative consumption of C_3 and C_4 plants closely reflects actual distributions on the ground, bison do not always forage indiscriminately. Particularly in regions where one of these photosynthetic types is only viable during a seasonally restricted period, bison diet is often dictated as much by the time spent focussing upon seasonally nutritious forage as by actual distribution. Although warm-season C_4 plants of the Canadian Plains account for an average of 20% of the available year round forage, they only become nutritionally attractive sources of food during the late summer when other sources desiccate and nutritional stress is high. Studies of the annual diets of bison in the Northern Plains indicate that while their relative consumption of different plant species is roughly equivalent to actual distributions, the vast majority of C_4 plants are consumed within a very restricted time period. Throughout the rest of the year preference for C_3 plants is not so much a result of an intensive focus but rather signifies a more uniform use of the landscape under conditions that are of an adequate nature to allow for the consideration of factors other than nutritional quality. Therefore, it is the behavioural responses of bison to the

seasonal patterns of forage production that ultimately dictate the composition of their diet.

In regions such as the Northern Plains where bison must intensify their use of one type of forage during a limited period, but are free to forage indiscriminately throughout the rest of the year, changes in the distribution of plants have a lesser role in determining diet than does the duration of climatic conditions associated with particular seasons. In the Canadian plains, bison primarily engage in selective foraging during the hot, droughts of the late summer. Under such conditions, the heat-adapted, drought-tolerant nature of C_4 plants makes them the most nutritious food choice available within the generally desiccated grasslands. In all other seasons, regardless of the level of dietary stress, the preferential consumption of C_4 plants provides little benefit and other compensatory behaviours must be employed. The ability of bison to subsist on a lower quality C_4 diet during the moisture deficient late summer is a key adaptation which has probably contributed to their historic success in the grassland biome. Furthermore, it suggests that during years which are inordinately hot and dry and consequently characterized by prolonged seasonal droughts, bison diets may show a significant increase in overall C_4 consumption.

These observations provide a basis from which to predict the dietary responses of prehistoric bison to various episodes of climatic change. Animals experiencing periods of hotter and drier climate over significant portions of their lives would have likely had to endure seasonal droughts of longer relative duration. As a result, their diets would be expected to reflect a proportionate increase in the consumption of C_4 grasses. Alternately, bison foraging during episodes of cool-aridity would derive little benefit by intensifying their use of either C_3 or C_4 plants. Such an environment would result in a reduction of nutritional quality across all forage species. Local herds would be forced to increase their range, becoming transient and potentially colonizing new areas with more nourishing vegetation. However, under conditions of adequate or abundant moisture, all vegetation reaches seasonal nutrition levels that are sufficient to limit dietary stress to within the tolerances of bison. Although the distribution of C_3 and C_4 grasses will change with respect to ambient temperatures in such an environment, the relative annual

consumption of warm or cool-season species is still dictated largely by the duration of the limited period in which C_4 grasses become attractive.

In summary then, although dietary behaviour may fluctuate in response to a variety of climatic events, an increase in selective foraging and a resulting change in the C_3 to C_4 plant ratio within the diet of Northern Plains bison, should primarily occur as a response to the specific stresses of a hot and dry environment. In general, this prediction appears to be borne out by the prehistoric data developed over the course of this project. Despite δD and $\delta^{15}N$ results indicating a series of distinct climatic episodes throughout the Holocene in southern Saskatchewan, there appears to have been little overall variation in the $\delta^{13}C$ composition of collagen from the various bison populations. The only cohort to show a significant dietary change was that of the Fitzgerald site. In conjunction with what appears to have been a comparatively hot and arid climate, collagen samples from the Fitzgerald bison show a significant increase in C_4 consumption. This dietary response corresponds to expectations for anatomically modern plains bison within such an environment. Furthermore, this event occurs within the context of the larger Scandic Climatic Interval, a period known to have been characterized by hotter and drier conditions.

The only prehistoric group which appears to deviate from expectations is the Norby site population. Despite a lifetime habitat with hot, dry climatic conditions that appear to have been analogous to those experienced by the Fitzgerald bison, the $\delta^{13}C$ results from the Norby population do not indicate a comparable dietary response. The apparent failure of the Norby bison to increase their reliance upon C_4 vegetation would have been a detriment to their overall success in an environment where the unique physiological advantages of these grasses provide a more nutritious alternative. The different dietary response of the Fitzgerald and Norby groups to similar environmental stimuli suggests a fundamental difference in contemporary foraging behaviour. Selective foraging for C_4 plants would have allowed the Fitzgerald bison to remain comparatively sedentary, mediating dietary stress through a more efficient use of local vegetation. This would have not only benefited individual fitness, but would have also allowed for the maintenance of higher population densities within a limited area. In contrast, the Norby bison would have had to become more transient in the search for

acceptable foraging patches and dependant upon the severity of the resulting stress, would have experienced varying levels of attritional mortality.

The causation of the behavioural differences that distinguish the Norby and Fitzgerald populations are likely complex, however there are at least two important possibilities. Although both populations lived during larger climatic episodes which are known to have been severe, the Altithermal interval which affected the Norby occupation, is widely acknowledged to have been of a intensity and duration that was otherwise unparalleled during the Holocene. It is possible that the associated desiccation of local forage and accompanying loss of coverage was of a magnitude which precluded a controlled response such as selective consumption. A number of studies have hypothesized that the biological and demographic changes which affected Mid-Holocene bison populations were likely a reflection of the harsh nature of the associated environment.

The data developed during this project concerning the Gowen site population may further contribute to such an interpretation. Although collagen samples from each of the three Altithermal period bison populations produced high $\delta^{15}N$ values consistent with heightened regional aridity, the δD measurements from the Gowen bison also indicated relatively low associated temperatures. Such an environment would have been particularly limiting to the nutrition and establishment of local vegetation and would have additionally amplified population stresses resulting from the already arid Mid-Holocene climate of the Northern Great Plains. Environmental changes during the Gowen period are also attested by more conventional archaeological analyses of the faunal assemblage. The intensive butchering practices employed by the human occupants of the site suggest conditions that required a somewhat different approach to subsistence. Whether this represents a more conservative approach to resource utilization or the preparation of a reliable staple such as pemmican, it appears that the local environment was exerting some type of stress. If episodes such as these were to have been a sporadic or even regular occurrence of the Altithermal interval, it is entirely foreseeable that circumstances may have ultimately expanded beyond the conventional tolerances of human and bison populations.

Alternatively, the dietary discrepancies of the Norby and Fitzgerald populations may reflect an adaptive biological difference. There has been an accumulation of fossil evidence indicating a substantial evolution of the genus *Bison* throughout the Holocene. It has been theorized that the ecology of the Early Holocene forms was primarily a reflection of physiological and behavioural adaptations that originated in response to the habitats of Pleistocene North America. As the grasslands of the modern Great plains began to develop in response to the warming and drying trends of the Holocene, bison came under pressure to adapt to the new cycles of the emerging vegetation. Although the resulting stresses initially reduced population sizes and forced social and geographic reorganization, they eventually engendered changes in physiology and behaviour that allowed bison to re-colonize much of the Great Plains during the Late Prehistoric. More specifically, anatomically modern bison are better equipped to tolerate diets with a high proportion of nutritionally poor xeric short-grass, allowing them to mediate the debilitating effects of the frequent warm-season droughts of the plains. Such an interpretation essentially posits that the climatic changes of the mid-Holocene were a catalyst for the evolutionary trends which resulted in the modern plains bison.

In any event, the demographic shifts among human and bison populations that are evident in the archaeological record of the Early, Middle and Late Prehistoric, bear witness to the influence of significant changes among a variety of ecological phenomena. Regardless of the exact causation, the diet differences of the Norby and Fitzgerald bison are important in that they represent the first direct evidence of a behavioural difference between Early Holocene bison and their Late Holocene counterparts. This finding, together with the environmental data developed through this study, helps both to characterize the unique ecological nature of the Northern Great Plains during the Middle Holocene and further to define the complex relationships between bison, humans and the Altithermal environment.

Further investigation will be required to determine whether Holocene climate changes ultimately forced the late Holocene development of specific behaviours, or instead caused their suppression during the Altithermal. Although isotopic analyses appear to have great potential for use in the investigation of prehistoric ecological relationships, specific problems will require attention. While stable hydrogen isotope

ratios in bone collagen show promise for use as a paleo-thermometer, quantitative analyses of past temperature regimes will require that the determining role of drinking water in the δD composition of tissues be defined and ultimately measured. It is also necessary that the discrimination between diet and bison tissue be quantified. The resolution of these issues may require the use of controlled feeding experiments similar to those which have been conducted to investigate issues related to the carbon-isotope composition of bison tissues.

In addition, more work is required to identify the exact mechanisms by which environmental aridity influences the stable nitrogen isotope composition of tissues from arid-land herbivores. Although the analysis presented in this study suggests that stable nitrogen isotope variation in the tissues of bison throughout the Holocene can be correlated with theoretical changes in regional aridity, more conclusive links between aridity and $\delta^{15}N$ values in bone collagen must be firmly established. Other investigations have also indicated a link between climate change and $^{15}N/^{14}N$ ratios in mammalian tissues. However, only a few studies have investigated the metabolic and physiological mechanisms by which climate determines the composition of an animal's tissue. These examinations have met with somewhat contradictory results and involved species which may not be directly comparable to large herbivorous mammals. Future investigations must employ either long-term studies of modern terrestrial herbivores from a variety of habitats or laboratory analyses in which water and protein intake are controlled. It is also possible that water and/or protein stress mechanisms are inadequate to explain the influence of climate on the $\delta^{15}N$ composition of mammalian tissues. If so, new theoretical models will be required.

Although stable carbon isotope analyses have had some success in application to ecological and archaeological problems, investigations involving bison populations should strive to adopt a regionally and temporally specific approach. While $\delta^{13}C$ values in the collagen of bison are directly related to the forage composition of their diets, the proportions in which they ingest these plant species appear to change in response to a number of criteria. Modern observations indicate that bison engage in selective consumption on a seasonal basis. This behaviour can not only confound the relationship between bison diet and local forage distributions, but can vary from region to region as

characteristics in the local environment change. Nevertheless, selective consumption does appear to occur as a predictable response to specific climatic phenomena. As long as there is an awareness of regional differences in both seasonal duration and the basic distribution of local vegetation, changes in diet may be predictable in response to shifts of specific climatic criteria.

Unfortunately, the results of this study appear to indicate that predictability, even within a regional context, is not constant through time. The dietary responses of bison to certain environmental patterns appear to be different earlier in the Holocene. As a result, more research must be directed towards identifying and understanding the regional and temporal variability of bison behaviour. Although certain ecological characteristics, such as vegetation distribution and forage quality, probably have an important influence upon behaviour regardless of geographic or temporal context, their specific impact in any given situation is complicated by other primary considerations including local climate and adaptive biology. However, stable isotope analyses may be uniquely suited to the investigation of such problems. As demonstrated, this technique can provide data concerning interrelated aspects of an animal's diet and environment. In addition, analytical substrates such as bone can be radiocarbon dated and may be used comparatively to examine physiological adaptations through time. Thus, the stable isotope analysis of archaeological bone provides a unique opportunity for detailed paleoecological investigations.

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Appendix A. A BRIEF REVIEW OF THE ARCHAEOLOGICAL SITES UTILIZED IN THIS THESIS

A.1 Introduction

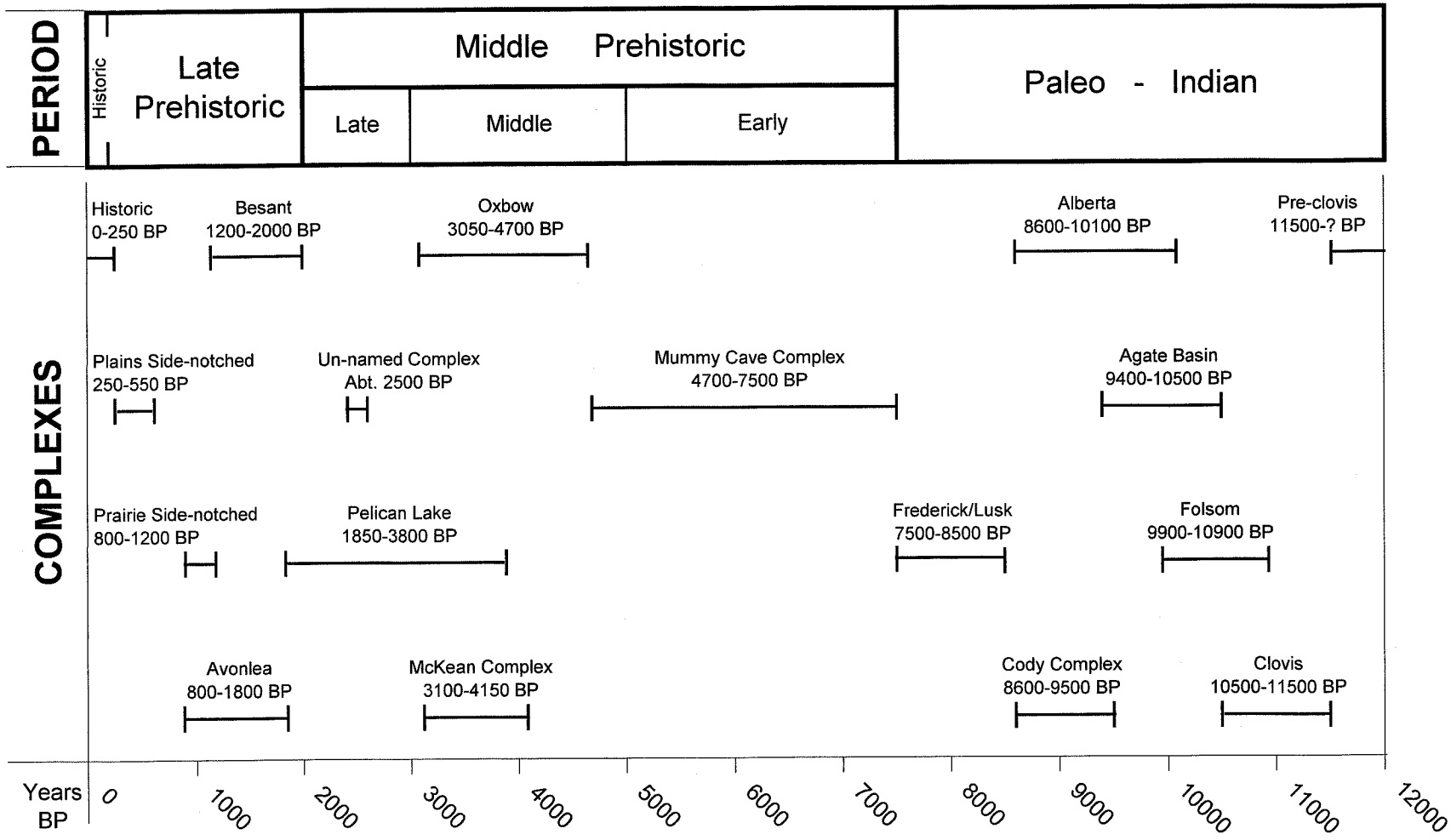
Eight different archaeological sites from southern Saskatchewan provided the bone specimens analyzed during this study. Each site had been previously excavated independently of this project and all of the recovered materials were subsequently under curation. Several criteria were applied during site selection. Foremost, it was important to select assemblages that were restricted to a specific region. The Saskatoon area was chosen because of its relatively high density of archaeological sites. For the most part, the sites included in this study occur within 40 km of each other, in and around the city of Saskatoon. Another consideration was to select sites that would cumulatively and evenly represent the time range of Saskatchewan prehistory. The inclusion of the Heron Eden site, located 200 km west of Saskatoon, was an attempt to extend the temporal range of this study into the early prehistory of Saskatchewan, a relatively poorly represented archaeological period in the province. Finally, those sites and cultural occupations that provided the highest bison MNI (Minimum Number of Individuals) assessments, were chosen in an attempt to generate the best possible interpretations of actual prehistoric bison populations.

Stratigraphic sections from some of the selected sites contained more than one cultural horizon. Bone samples were only taken from those components that met the aforementioned criteria and which provided an opportunity to examine a desirable time period and/or associated culture complex. Classification and description of culture history in Saskatchewan and the Northern plains has been an evolving process and as such, several different classification schemes exist. Linnamae *et al.* (1988) present a synthesis of the paleoculture sequence for Saskatchewan, based on Mulloy's (1958) earlier work. This synthesis is largely tailored to the Saskatoon area, and as such, is the one used here (Figure A1). The terminology concerning the associated time periods

Has, however, been updated to reflect Walker's (1992) recent synthesis for Saskatchewan. The age ranges associated with the various time periods and cultural complexes are largely derived from radiocarbon dates and should be considered to be somewhat generalized. As previously stated, culture history is a constantly developing phenomenon; new finds and better dating techniques can potentially reshape its interpretation.

The following is presented as a brief review of each of the sites from which materials utilized in this thesis were obtained. It is not meant to be comprehensive, and in each case, the source analyses are referenced extensively. For those sites that contained more than one cultural component, emphasis has been placed upon only those horizons that yielded materials analyzed in this study. Where warranted, a brief introduction to the specific areas at which some of the sites are located has also been provided.

Figure A1. The Paleocultural Sequence For Southern Saskatchewan (Adapted From Dyck 1983; Linnamae *et al.* 1988 and Walker 1992).



A.2 Southwestern Saskatchewan

A.2.1 The Heron Eden Site (EeOi - 11)

The Heron Eden site represents the northernmost excavated Cody Complex age site on the Great Plains. It is located in Southwestern Saskatchewan approximately 13 km south of Prelate (Corbeil 1995). The site was originally discovered in 1973, on the surface of a cultivated field by avocational archaeologists Ruth and Fulton Heron. In 1987, members of the Saskatchewan Archaeological Society and archaeologists from the Royal Saskatchewan Museum, visited the site (Corbeil 1995). Excavations were begun in the summer of 1989 by members of the southwestern chapter of the Saskatchewan Archaeological Society under the direction of Dr. Urve Linnamae from the University of Saskatchewan (Corbeil 1995). Investigations continued until 1992. A total of 82 square meters were eventually excavated at the site (Corbeil 1995). All materials recovered during excavation of the Heron Eden site are currently held in storage at the curation facility within the Wanuskewin Heritage Park Archaeological Laboratory.

The Heron Eden site is represented by a single occupation within a paleosol occurring immediately below the plow zone. The full extent of this paleosol was difficult to assess as cultivation and sedimentary deflation had disrupted the areas north, west and south of the excavations (Corbeil 1995). Nevertheless, a portion of the bone bed remained intact within the area of excavation. Of the 14 projectile points recovered, only eight were identifiable. Three of these, identified as Scottsbluff type points, were found in situ. The remaining five points were collected on the surface of the site and were determined to represent four Scottsbluff points and one Eden point (Corbeil 1995). Five un-calibrated radiocarbon dates were obtained from unburned bison bone samples. While two of the dates were discarded, the remaining three clustered in age around 9,000 years BP (Corbeil 1995).

The Heron Eden artifact assemblage also contained several lithic categories other than projectile points (Corbeil 1995). Identified tools included a bifacial chopper, six end scrapers, a burin, several re-touched and/or utilized flakes and a few unifacial tools. Raw materials utilized in the manufacture of all lithics from the site comprised a mixture of both local and exotic sources. These included jasper, chalcedony, fused

shale, quartzite, chert, agate, silicified wood, Knife River flint and possibly Beaver River sandstone. Debitage was also recovered during the excavations and included a variety of flakes and shatter. The proportions of identifiable flake types seemed to indicate a concentration on tool repair, sharpening and rejuvenation rather than primary tool manufacture. It was thus, concluded that the Heron Eden lithic assemblage resulted from a special purpose activity, such as bison carcass processing, rather than a multi-activity campsite (Corbeil 1995).

The faunal assemblage from the Heron Eden site consists primarily of bison bone. Only 11 non-bison specimens were recovered from the occupation including two canid (wolf) metacarpals and one pronghorn astragalus (Corbeil 1995). Of the 220,164 bone specimens recovered form the site, roughly 90% was unidentifiable reflecting the high degree of fragmentation that occurred at the site (Corbeil 1995). Using mandibular third molar counts, the MNI (Minimum Number of Individuals) for bison at the site was 37; using fourth carpals, this measure was reduced to 36 (Corbeil 1995). Several techniques of sex determination were employed including bivariate plots of carpal/tarsal measurements and long bone measurements, as well as discriminant function analysis of metapodial elements. Each of these lines of evidence seem to separately and cumulatively indicate a mixed sex composition for the assemblage including relatively equal numbers of males and females/immature bison (Corbeil 1995).

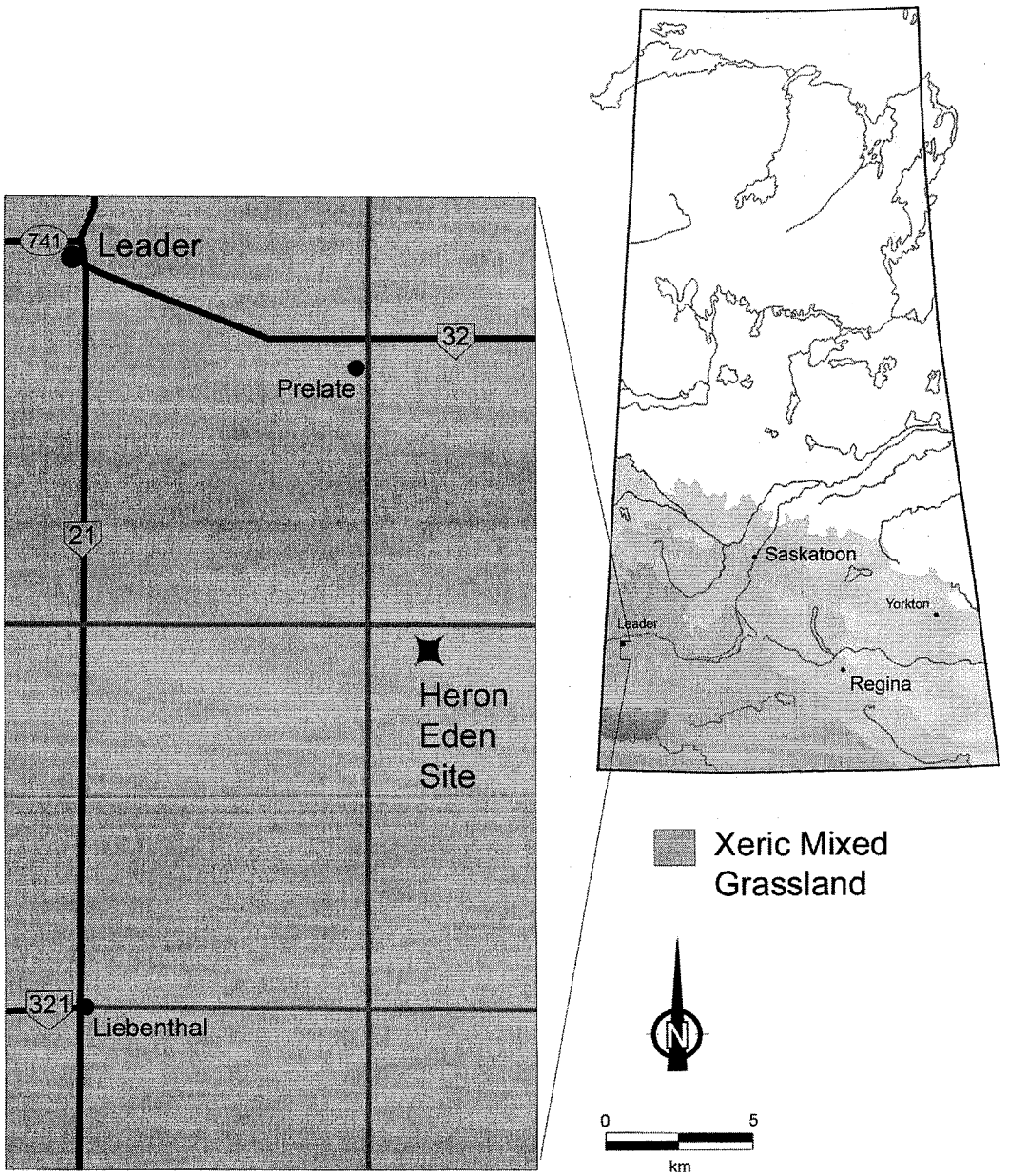
Dental eruption sequences, tooth wear patterns, measures of molar metaconid height, exostylid wear patterns and positioning of enamel-root lines relative to the alveolus were all techniques employed to infer the ages of the animals. Comparisons of the data recorded for the Heron Eden bison with that from other archaeological bison populations seemed to indicate relatively constant age increments within the Heron Eden bison. When gauged against the restricted April/May birthing season of bison, The Heron Eden kill was considered to have taken place in January/December (Corbeil 1995). The bison age and sex data indicate the presence of a well-mixed assemblage of individuals. If the seasonality assumptions that were made are correct, then male and female/immature herds should have been separate at the time of the kill. Corbeil (1995) suggests that the Heron Eden site may well represent at least two distinct kill events in succession at the site (Corbeil 1995). The high number of individuals at the site and the

highly fragmentary nature of the recovered specimens suggest that the site may represent a kill/butchery locality (Corbeil 1995).

The age of the Heron Eden assemblage necessitated that an attempt be made to determine the probable species from which the bison remains were derived. Due to a lack of un-fragmented cranial elements, only post-cranial measurements could be made. These data were then compared to previously determined values obtained from other similarly aged archaeological bison assemblages. Generally, Heron Eden females and immature bison fell within the ranges noted for both *Bison bison occidentalis* and *Bison bison antiquus*. Males, on the other hand, rated larger than the expected values for both fossil forms. On the basis of this determination, the Heron Eden bison have been determined to represent a *B. b. antiquus* form (Corbeil 1995:70-71).

Generally, the Heron Eden faunal assemblage exhibits a relatively high degree of weathering which obscures evidence of cultural modification to the bone. A lack of evidence suggesting carnivore and/or rodent modification may also result from this (Corbeil 1995:118). Most specimens exhibited a high degree of fragmentation. Elements and elemental portions represented correlated highly with density indices while utility indices represented no meaningful correlation (Corbeil 1995:117). Corbeil (1995) concluded that fragmentation at the site was likely due to attritional processes and density dependant destruction. Despite this, analysis of the lithic assemblage, the fragmentary nature of remains and an accounting of the number of bison recovered during excavation suggest that the Heron Eden site represents a Cody Complex kill/butchery site (Corbeil 1995).

Figure A2. Location of the Heron Eden Site in Southwestern Saskatchewan



A.3 The Saskatoon Region

A.3.1 The Harder Site (FbNs –1)

The Harder Site was discovered in June of 1969 by Ian Dyck after noticing cultural debris on the crown of a newly bladed north-south road (Dyck 1977, Morlan 1994). Test excavations began in the late summer of that year and helped to characterize the site and define its extent. It is located about 23 km west and 10 km north of Saskatoon and occurs in a shallow dune depression near the north edge of the Dunfermline Sand Hills. Dyck named the Harder site after a family living near the excavations (Dyck 1977). Intensive investigations were undertaken over three field seasons (1970 – 1972) and resulted in the excavation of over 130 m^2 (Dyck 1977, Morlan 1994). All materials recovered from the site are currently curated at the Royal Saskatchewan Museum in Regina. In addition to the main excavations, an east-west profile trench extending about 67 meters was excavated with a backhoe to help define the spatial extent of the site. North-south profiles were available from the ditch running parallel to the new road. Dyck (1977) identified one cultural occupation at the site occurring within a zone of gray-black fine textured sand. The level varies in thickness between about 12 to 28 cm and occurs roughly parallel to the modern surface between about 30 to 60 cm BS. The upper and lower extents of the layer were determined to be transitional and gradually blend into the surrounding levels (Dyck 1977).

Dyck (1977) obtained two radiocarbon dates from fragments of charred comminuted bone. The dates were isolated from the extracted collagen of each sample (Dyck 1977). The un-calibrated samples dated to 3,360 ± 120 and 3,425 ± 105 R.C.Y. BP respectively (Dyck 1977). Dyck (1977) notes in his analysis that the Harder site age is about 1000 years younger than the range suggested by several other Oxbow component dates. He explains this discrepancy by citing comparable ages from two other sites in the Dunfermline Sand Hills (Moon Lake and Carruthers) and by suggesting a much longer survival of the Oxbow complex than was previously inferred (Dyck 1977, Morlan 1994). Several years later, Morlan (1994) re-examined the Harder site collection and submitted three samples of bison bone for radiocarbon dating. He hypothesized that the younger original Harder dates may have resulted in part through the use of inadequate pretreatment methods during the dating of Dyck's original

samples. The three un-calibrated dates obtained by Morlan (1994) are as follows: 3,420 ± 140, 4,410 ± 150 and 4,190 ± 90 R.C.Y. BP. The youngest of these dates fits with the original dates for the site. The other dates, however, yielded ages roughly a millenium older, bringing the age of the site closer to the range typically assigned to Oxbow culture.

While Morlan (1994) acknowledges the possibility that the Harder site may contain more than one component, he concludes that a more likely explanation for these discrepancies is potential contamination of some of the Harder site bones. He notes that during his analysis some of the bone appeared to have a porcelain-like texture and some seemed to be enriched and/or coated with a clay mineral. Some time after initial excavation, Ian Dyck returned to the Harder site with a geochemist and they concluded that most of the faunal assemblage occurs in a zone of groundwater discharge and thus, may have been subject to geochemical alteration (Morlan 1994). Despite an improvement in methods Morlan (1994) admits that he is unable to explain why the contamination evidenced in the younger of the new dates is immune to pretreatment. In the end, both Morlan (1994) and Dyck (1977) seem to agree that the Harder site contains a single cultural component. Morlan (1994) suggests that the two older dates likely provide the best estimate of the Harder site age.

Highly utilized and re-worked projectile points and end scrapers dominated the Harder lithic assemblage (Dyck 1977). Twenty-four nearly complete side-notched (Oxbow) projectile points were recovered along with identifiable fragments from 48 other side-notched points. Other stone tools included small uniface knives, point pre-forms, perforators, a few large bifaces, a few retouched flakes and several coarse stones used as hammers, anvils and cooking stones. Stone debitage (debris) totalled 3,874 pieces and included a variety of flakes, shatter, cores and core fragments. Petrified wood and various cherts were the primarily utilized raw materials; but other types including quartzites, chalcedonies and a selection of quartz, basalt and fused-shale sources were also used (Dyck 1977). Six bone tools (including antler) were also recovered from the Harder site.

The faunal assemblage was dominated by bison, but also included a sampling of other species (Dyck 1977). Morlan's (1994) re-examination of the Harder site faunal

assemblage re-assigned a few of the taxa identified by Dyck (1977) and is thus, used here. Based on general size, but also with consideration of several specific post cranial measurements, Morlan (1994) assigns the Harder site bison to the modern species *Bison bison bison* (Plains Bison). Specimens of wolf, fox, coyote, moose, rabbit, fisher, badger, bird and deer were also recovered at the site (Morlan 1994). The highly fragmented nature of the bison remains made analysis somewhat difficult but Dyck (1977) suggested an MNI of 17 individuals based on fragments of distal left tibia. This fragmentation also suggested a high degree of processing and utilization of the remains. This, in addition to a somewhat selective representation of elements and small overall amounts of bone, suggested that the Harder locality may have been a campsite (Dyck 1977). Dyck (1977) identifies several "dwelling floors" and "outside activity areas" through analysis of the Harder sites single hearth, smudge pits, refuse areas and through the identification of areas of differential artifact concentration. He suggests that this evidence provides ample confirmation of a campsite. Morlan's (1994) re-analysis suggests that element representation at Harder does not seem to reflect typical campsite utility concerns. He, however, argues that this is probably evidence of human processing and differential survivorship, and support's Dyck's campsite interpretation. Harder is thus, one of the largest known Oxbow campsites (Morlan 1994).

Dyck (1977) initially suggested that Harder was likely a winter campsite due to a lack of locally available water. Winter would thus be the only time that a source of readily accessible fresh water, in the form of snow, would be available. Morlan (1994) confirmed this interpretation examining dental eruption patterns in the available teeth from the Harder site bison. Two Harder specimens appeared to have died at about the age of seven months. Assuming that birth occurred in the spring, death may be inferred as to have occurred in the winter (Morlan 1994). Fracture patterns amongst some of the bones suggested that limbs may have been frozen prior to processing. Winter would have provided an opportunity for frozen storage of surplus meat (Morlan 1994). Finally, Morlan's (1994) analysis also suggests that the sex and age composition of the bison assemblage does not represent a single mass-kill event. Bulls and cows are almost equally represented. Combinations of communal and solitary hunting methods likely account for the mixture of individuals present (Morlan 1994).

A.3.2 The Fitzgerald Site (ElNp - 8)

The Fitzgerald site, "named for the owners of the bison paddock where the site is located" (Hjermstad 1996:1), was excavated during the summers of 1992 and 1993 by staff and students from the Department of Anthropology and Archaeology at the University of Saskatchewan. The site occurs roughly 15 km southeast of Saskatoon, within the Aspen Parkland ecoregion approximately 20 km north of the present day boundary with the Mesic Prairie zone (Hjermstad 1996:8). The site was discovered in the spring of 1991 by the landowner and was subsequently reported to researchers at the Department of Anthropology and Archaeology. The investigators confirmed the presence of an intact cultural component located within a roughly 15 cm thick paleosol approximately 50 cm below surface (Hjermstad 1996). All materials, subsequently recovered during excavation of the site, are currently held in storage at the Department of Anthropology and Archaeology at the University of Saskatechewan.

The 1992 field season began with 10 weeks of survey designed to determine the vertical and horizontal extent of the site, as well as the nature of the occupation (Hjermstad 1996). Testing suggested that cultural materials were located within a single 20 to 30 cm thick layer of brown paleosol beneath 75 cm of fine grained yellow sand. Furthermore, two separate activity areas were indicated. Area 1 was believed to represent a main kill area, while Area 2 appeared to be the site of secondary processing (Hjermstad 1996). By the end of the 1993 field season, a total of 42 m^2 had been excavated in the main kill area (Area 1) and 31 m^2 had been excavated in the processing area (Area 2) (Hjermstad 1996). 68 of the 143 complete and fragmented projectile points that were recovered were identified as Besant style points composed almost exclusively of Knife River Flint. The cumulative assemblage suggested that Fitzgerald represented an undisturbed Besant pound and processing area (Hjermstad 1996). Four radiocarbon dates were obtained from the acid soluble collagen fractions of bone samples collected from the site. Calibrated, the four dates cluster roughly around 1,300 years BP (Hjermstad 1996). These dates are consistent with the Besant style points recovered during excavation. The assemblage is therefore, considered to represent a later Besant occupation (Hjermstad 1996).

Other than projectile points, there were only 22 formed tools recovered from the Fitzgerald site (Hjermstad 1996). Tools from the kill area included end scrapers, a small biface, two unifaces, and a pièce esquillèe. 62 utilized flakes were also recovered from the kill area. 16 formed tools were found in the processing area and included several end scrapers, a side scraper and four unifaces. 68 utilized or retouched flakes were also recovered from the processing area. 2,030 pieces of debitage were collected from the Fitzgerald site in total (Hjermstad 1996). This count includes cores, flakes and shatter. The majority of the debitage was discovered in the processing area. Both the kill and processing areas were found to be largely devoid of core fragments, shatter and primary flakes suggesting that a lot of retouch and re-sharpening, and little tool manufacture, was occurring at the Fitzgerald site. The large number of expedient tools would seem to support this conclusion. The lithic artifacts at Fitzgerald were largely constructed of Knife River flint, a material commonly associated with Besant culture (Hjermstad 1996). Other materials included locally available chert, quartzite, chalcedony, siltstone, jasper, silicified peat and petrified wood; while exotic materials included fused shale, obsidian and Tongue River silicified sediment (Hjermstad 1996).

The Fitzgerald site also provided a small collection of ceramics and bone tools (Hjermstad 1996). Three ceramic shards were recovered from both the kill and processing areas. They did not, however, prove diagnostic (Hjermstad 1996). Only two tools were identifiable from amongst the various bone fragments recovered at the site. These were interpreted to be a bone needle and a scraping tool. Five bone fragments, tentatively identified as decorative items were also collected (Hjermstad 1996). Approximately 16 site features were also described during excavation. Features found in the kill area included post molds, bone uprights and ash with burned soil stains. These features, when considered alongside the recovered artifacts, suggested that a prehistoric pound may have been the method of bison procurement that produced the kill area assemblage (Hjermstad 1996). Features found in the processing area included a basin shaped pit and an area of densely packed bone uprights. Again, these features in conjunction with the recovered tool and debitage, would seem to verify the interpretation of Area 2 as a processing locality (Hjermstad 1996).

The identifiable faunal assemblage obtained from the Fitzgerald site was solely composed of bison and consisted of a NISP (Number of Identifiable Specimens) of 11,287 weighing 492,818 grams (Hjermstad 1996). MNI counts indicated a minimum of 49 individuals represented at the site (Hjermstad 1996). Although no specific attempt at analysis was made, the Fitzgerald site bison were assumed to represent the modern form of plains bison (*Bison bison bison*). Demographic reconstructions, utilizing a variety of age and sex determination techniques, seemed to indicate a number of unusual features. The identifiable assemblage appeared to be composed of relatively equal numbers of adult males and females, notable because bison herds are largely segregated throughout most seasons of the year. Age determination techniques indicated a restricted age composition, indicative of a kill that may have taken place in the late fall; not a time of the year during which sex specific herds are known to aggregate (Hjermstad 1996). Hjermstad (1996) suggests that the Fitzgerald assemblage may thus, have resulted from numerous separate kill episodes within a single season.

Another unusual feature of the Fitzgerald site faunal assemblage was that the reconstructed age profiles for the site population seemed to indicate an unusual number of older animals (Hjermstad 1996). Taphonomic analyses seemed to show that while the assemblage was heavily butchered leaving few complete elements or articulations, other processes such as carnivore chewing, rodent gnawing, geologic transportation and weathering had minimal impact on element representation and survivorship. There was therefore little to indicate that element counts had been severely altered by anything but cultural activities (Hjermstad 1996). Hjermstad (1996) suggests that the surprising proportion of adult bison in the assemblage may be an indicator of favourable environmental conditions which would act to promote health within the local herds. He proposes that an environment moister than today's would create conditions favourable to grassland productivity and thus, the sustenance of larger herds and longer lived animals. Such a scenario could then be used to explain the observed proliferation of the large herd dependant communal hunting strategies evidenced during Besant times (Hjermstad 1996).

A.3.3 The Tschetter Site (FbNr – 1)

The Tschetter site is located approximately 16 km west and 10 km north of Saskatoon on the eastern margin of the Dunfermline Sand Hills (Walker 1979, Linnamae 1988). The site had been known for some time by the Tschetter family upon whose land it was excavated, but did come to the attention of archaeologists until 1970 when the road that bisects it was being improved (Walker 1979, Linnamae 1988). The site was tested in 1971 and subsequently excavated between 1971 and 1976 by students from the University of Saskatchewan archaeological field school under the direction of Dr. Urve Linnamae (Linnamae 1988). At this time a total of over 160 square metres were excavated from the site. Additional investigations occurred in 1979, 1980 and 1984 and resulted in several more square metres of excavation (Linnamae 1988). All of the recovered materials are currently curated at the Department of Archaeology at the University of Saskatchewan. The site itself consists of a bonebed located within a single cultural horizon. The bone bed occurs within a dark loam beneath a sandy root mat and a sand layer which together are roughly 20 to 30cm in thickness. The maximum thickness of the bonebed is approximately 35cm. Its spatial dimensions were estimated at approximately 3,500 square meters, with an east-west length of 70m and a north-south run of 50m (Linnamae 1988).

Three un-calibrated radiocarbon dates have been determined for the site from bone collagen. These ages are 1,005 ± 75, 1,020 ± 100 and 915 ± 45 R.C.Y. BP respectively. Linnamae (1988) considers these dates to be roughly contemporaneous and thus, suggests that they provide a good estimate of the age of the Tschetter site. Projectile points recovered from the excavations provided additional support for this hypothesis. Roughly 270 complete and fragmented projectile points, comprising nearly 55% of the total lithic assemblage, were collected. All points that were identifiable were consistent with the Prairie Side-notched form of the "Old Women's" culture complex (Linnamae 1988). This point type has been consistently dated to between about 800 and 1,200 years BP. In addition, pottery fragments representing four different vessels were also found at the site. Although this pottery was not attributable to a particular tradition, morphological similarities to shards recovered from other Late prehistoric sites allowed the Tschetter site ceramics to be generally reconciled with the site age as determined by radiocarbon dates and point morphology comparisons (Linnamae 1988).

In addition to projectile points, the lithic assemblage also included a variety of tools and debitage. A significant number of bifacial and unifacial tools were recovered at the Tschetter site and probably indicate that cutting and butchering were prevalent activities at the site (Linnamae 1988). Expedient tools such as utilized flakes were also identified and lend support the idea that significant cutting work was being undertaken. End scrapers recovered from the site suggest that hide preparation was also occurring (Linnamae 1988). In addition to meat and hide processing, a handful of large stone tools including quartzite choppers and a large maul attest to the breaking and processing of animal bone as an important activity. Perforators, drills and "spokeshaves" (used to shape and straighten shafts) were also identified in small frequencies (Linnamae 1988). The raw materials used to fashion these tools were largely local in origin. Swan River Chert, South Saskatchewan River Chalcedony and various pebble cherts comprise over 70% of the materials used. Other sources, including petrified wood, white chalcedony, jasper, quartz, quartzite and basalt, may have been locally obtained but could also have been brought from farther abroad (Linnamae 1988). This suggests that the group using this site may have ranged somewhat from the site area, or that several groups may have utilized the site. An extremely small sample of more exotic materials (obsidian and Knife River Flint) demonstrate that these peoples maintained trading ties to distant areas, but the limited occurrence of these materials also highlights the difficulty of obtaining them (Linnamae 1988).

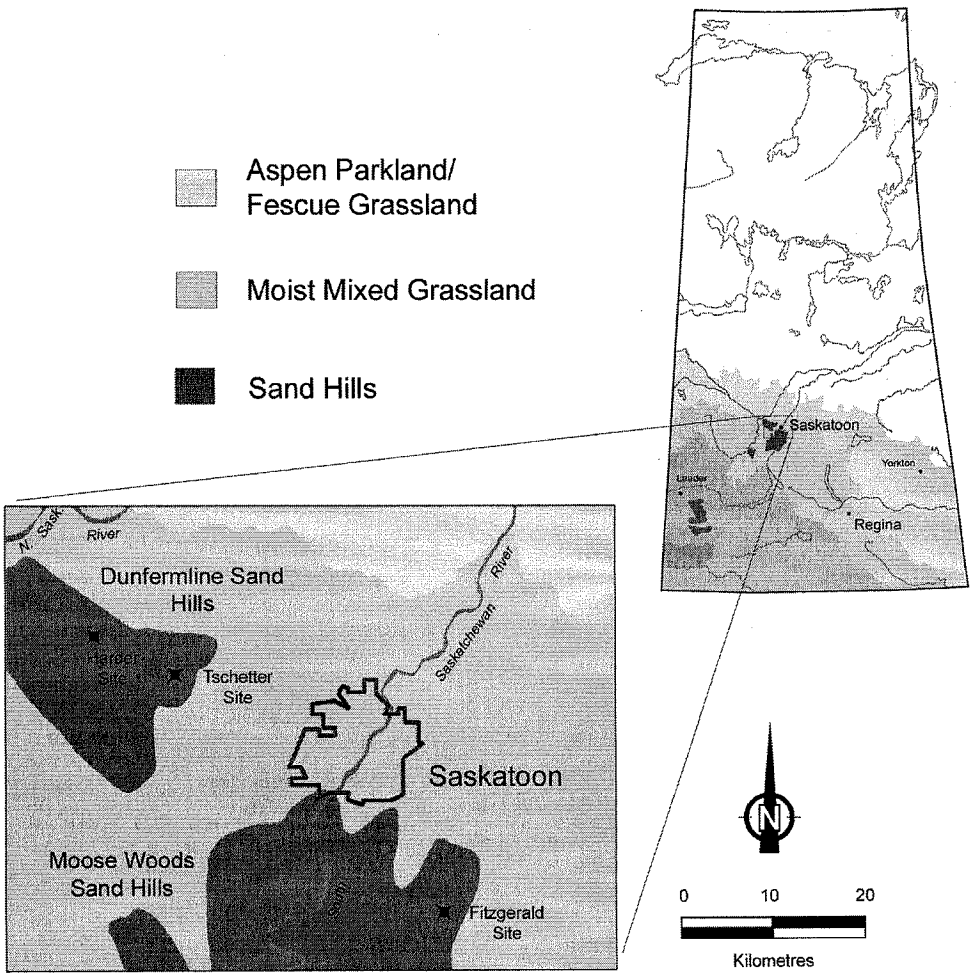
The faunal assemblage at the Tschetter site was dominated (over 99%) by bison (Walker 1979). The few non-bison remains represent badger, skunk, hare, various canids and unidentifiable bird remains. Intrusive species included mice, voles and ground squirrel. An MNI of 86 bison was determined for the Tschetter site based upon mandibular third molars (Walker 1979). The estimation has since been revised, through continued excavation, to 93 (Linnamae 1988). The highly fragmented nature of the assemblage, under-representation of some elements (particularly long bone specimens) and identification of specific fracture patterns, led to speculation that marrow extraction was a key activity at the site (Walker 1979, Linnamae 1988). In addition to this focus, bones were used to fashion several different types of tools. Walker (1979) identified 135 bone tools in the Tschetter assemblage. These included various perforators,

handles, knives and spatulates as well as tools fashioned for chopping, flint-knapping, hide-scraping, and polishing. Other miscellaneous tools showed evidence of utilization, but little to indicate function (Walker 1979). An interesting pathology noted by Walker (1979) during his analysis was trauma caused to a vertebral process by an imbedded projectile point, and subsequent bone re-growth suggesting that the source animal had evaded other hunters prior to the events at the Tschetter kill site.

Walker (1979) also examined the demography of the Tschetter site bison. These animals are assumed to represent the modern form of plains bison (*Bison bison bison*). Despite the small number of specific bones from which sex determinations could be made, Walker (1979) was able to determine that all but one of 16 suitable individuals were probably female. This determination was based upon mandibular measurements. 13 immature and 27 mature specimens were suitable for age determination analyses. Dental eruption and wear patterns were used to assign each sample to an age grouping. The reconstructed age profile for all bison suggested that the kill events occurred sometime during the winter (Walker 1979).

The identification of eight apparent post-holes during excavation of the site, led to its interpretation as a probable pound site (Linnamae 1988). A corral structure would have allowed for the collection and slaughter of several bison over an indefinite period of time. The presence of the Tschetter site bonebed would seem to indicate some form of communal bison procurement. Linnamae (1988) suggests that the significant number of recovered bison and the presence of post-holes, lends credence to this interpretation. In addition, ethnographic data support the frequent use of pounds during winter in areas peripheral to the plains (Linnamae 1988). Other features indicate that secondary processing activities were also occurring at the locality. These include two pits, into which bone was apparently sorted, and several charcoal and ash concentrations that are thought to have been associated with cooking or bone-grease extraction. An unusual feature consisting of several bone uprights was also documented, but not interpreted (Linnamae 1988). Nevertheless, the occurrence of these secondary activities does not contradict the interpretation of the Tschetter site as a winter bison pound.

Figure A3. Archaeological Sites in the Saskatoon Region



A.4 The City of Saskatoon

A.4.1 The Gowen Sites (FaNq - 25 and FaNq - 32)

The Gowen sites, named after their discoverer Charlie Gowen, are located on a major terrace of the South Saskatchewan River, within the boundaries of the City of Saskatoon. Their position on the terrace is approximately 800 metres west of the river, in the southwest edge of the city at the site of the Saskatoon City landfill (Walker 1992). Gowen 1 (FaNq - 25) was first discovered in 1977 as city workers excavated a large borrow pit in the landfill. The site and some of the recovered artifacts were brought to the attention of staff at the Department of Anthropology and Archaeology at the University of Saskatchewan. Members of the department, with the assistance of local Saskatchewan Archaeological Society members, began and finished excavations later that year (Walker 1992). Gowen 2 (FaNq - 32) was similarly discovered by work crews in 1980. The new site was located roughly 70 meters west of the original. Investigations were begun that same day by departmental archaeologists and were completed by the end of the 1981 field season (Walker 1992). Cultural deposits at both sites were localized to a single paleosol (Walker 1992).

A total of 98 projectile points were recovered from the Gowen sites; 23 from Gowen 1 and 75 from Gowen 2 (Walker 1988). Of those points identified, styles included Mount Albion Corner-notched, Bitteroot Side-notched and Blackwater Side-notched (Walker 1988). All of these fit into expected styles for the Mummy Cave complex. Gowen Side-notched points were a new style designation first described by Walker (1992) in reference to a new point style identified initially in the Gowen 1 assemblage. He concluded that Gowen points fit within the side-notched tradition that characterized the Mummy Cave complex. Five radiocarbon dates were taken from Gowen 1 and four were obtained from Gowen 2 (Walker 1992). While the overall range of these un-calibrated dates span a period from 4,700 to 6,200 radiocarbon years ago, eight of the nine dates clustered around 6,000 radiocarbon years in age (Walker 1992). This average date fits well with established Mummy Cave projectile point chronology.

The cultural assemblage from Gowen 1 included 226 formed tools, while the Gowen 2 assemblage was somewhat more extensive and produced about 350 (Walker 1992). Aside from finished projectile points, these totals were comprised of preforms,

hafted bifaces, bifacial knives, unifaces, side scrapers/end scrapers, spokeshaves, gouges, perforators, hammer/anvil stones and a variety of utilized and/or retouched flakes. The Gowen 1 debitage consisted of 3,767 artifacts, primarily flakes, and 24 complete and fragmentary bipolar cores. Gowen 2 provided 12,395 pieces of debitage (flakes and shatter) and 66 complete cores and 43 fragmentary cores. A large quantity of fire-cracked rock was also recovered from both areas (Walker 1992).

Similarities amongst the lithic assemblages from both sites would tend to give credence to the argument that the Gowen sites are chronologically contemporaneous. They may, in fact, represent two different activity areas from the same site (Walker 1992). The most commonly utilized lithic materials were cherts and quartzites of various grades and colours. Chalcedony, petrified wood, limestone and sandstone were other locally available lithic materials that were discovered at Gowen 1. More exotic materials included various agates and Knife River flint (Walker 1992). In addition to fashioned lithic artifacts, several bone tools were recovered from both sites. Most were created from bison bone, but bird, pronghorn and canid remains were also utilized (Walker 1992).

The faunal assemblage from Gowen 1 was dominated by bison remains, but also included a variety of canid elements and at least six pronghorn specimens (Walker 1992). Several small rodent elements and a right first phalanx from a crow were also identified (Walker 1992). Gowen 2 was similarly composed, but overall was a somewhat larger collection. The most remarkable feature of the faunal assemblage from the Gowen sites was its highly fragmented state. The association of these remains with at least two anvil stones, gives the impression that the maximum utilization and processing of available animal resources occurred (Walker 1992). Speciation of the Gowen site bison was made more difficult by the fact that no complete crania or horn cores were recovered from the site. Measurements of carpal and tarsal elements suggest that Gowen site bison fit within a larger size range than that assigned for modern bison. This suggests that the Gowen bison may represent an extinct subspecies (Walker 1992).

The Gowen 1 bison assemblage consisted of 114 specimens of bone. A minimum of seven individuals were represented through tarsal counts, and two more immature specimens was indicated by the presence of un-fused cervical vertebrae

(Walker 1992). Gowen 2 contained a bison collection with an NISP of 217. At least 9 individuals were represented through carpal counts (Walker 1992). The Gowen 2 bison assemblage had almost twice the number of identifiable specimens (217) and represented at least 14 individual animals. Mandibular and maxillary molar eruption and wear patterns from Gowen 1 bison were used to estimate the age profile of the associated bison population. A restricted age sample was revealed suggesting that the kill event associated with the site occurred during the late summer. Seasonality may be inferred for Gowen 2 from Gowen 1 because both are considered to be contemoraneous. Nevertheless, Walker (1992) cautions that inferring seasonality for the Gowen 2 assemblage may only be considered tentatively due to the fragmentation of the available remains and a lack of immature animals from which to draw definite conclusions (Walker 1992).

Features identified at Gowen 1 included a series of pits and hearths that may have been associated with a variety of activities including bone and hide processing. Gowen 2 was found to possess eleven identifiable features and included ash lenses, bone piles, soil stains, a selection of pits and hearths, and an area of concentrated lithic debris that included several post-holes (Walker 1992). Evidence from Gowen 2, including the presence of post-holes and the possible identification of the outline of a living mat, suggests that the area may have been associated with habitation activities. Generally, the sites are considered to represent a short duration bison hunting camp and a temporary processing area. Artifact recovery and excavation sampling at the sites were greatly assisted via flotation of soil samples. Flotation allowed for the recovery of small, carbonized seeds. The identification of *Chenopodium* seeds, which are generally available in summer or autumn, is considered to be consistent with seasonality determinations at the site (Walker 1992). Alternatively, the relative paucity of these remains may suggest the occurrence of adverse environmental conditions at the time of occupation (Walker 1992).

A.4.2 The Norby Site (FaNq - 56)

Les Norby discovered what is now the Norby site in 1988 during excavations for a new residential basement. The site is located within the city of Saskatoon in the 900

block of Avenue M South (Zurburg 1991). Mr. Norby contacted the Department of Anthropology and Archaeology at the University of Saskatchewan and researchers were dispatched to examine the site. Test excavations were carried out in the fall of 1988 and full-scale excavations were begun in the spring of 1989 (Zurburg 1991). Students from the Anthropology and Archaeology department field school, along with students from King George Elementary School and a sporadic group of volunteers, completed investigations at the Norby site by the end of the 1989 field season. "The Norby site profiles were fairly straightforward because there was only one occupation level to contend with" (Zurburg 1991:39). The top 30 cm of the profile were largely disturbed. The occupied horizon began at roughly 110 cm BS and ended once a depth of 120-125 cm had been reached (Zurburg 1991). Excavation occurred in four localized areas labeled A to D, and a total area of 50 square meters was eventually removed (Zurburg 1991).

Four complete projectile points and two point fragments were recovered from the site. All of the complete points were found in situ and are thought to represent three Gowen side-notched points and one Manitoba point (Zurburg 1991). The three Gowen points suggest an Early Middle Prehistoric age for the occupation while the recovery of a Manitoba point may suggest an earlier age. Although Manitoba points have been associated with Paleoindian assemblages in the USA, Zurburg (1991) suggests that the Norby site discovery indicates that the association of the Manitoba style with the Paleoindian complexes may have been a premature assumption. The Manitoba form may in fact, have had a later expression in Canada. It may also suggest that Norby is composed of more than one cultural occupation, although Zurburg (1991) discovered little evidence of this. Three radiocarbon dates were obtained from bison bone samples and all three un-calibrated dates clustered around 5,700 years BP placing the occupation as roughly contemporaneous with other Mummy Cave complex occupation sites (Zurburg 1991).

The Norby site lithic assemblage appears to be uncharacteristically small for a bison kill site (Zurburg 1991). While only 20 formed tools were recovered, those tools and debitage that were documented suggest that primary butchering was carried out at the site (Zurburg 1991). This interpretation becomes more evident when considered

alongside the site's faunal collection. Aside from projectile points, recovered tools include two point preforms, five bifacial tools of indeterminate function, ten uniface fragments and a large granite rock, possibly used as a hammer/anvil. 176 pieces of debitage, primarily flakes, were also found (Zurburg 1991). Swan River chert was the most abundant material type, comprising of 60 percent of the sample. Other local materials included silicified peat, silicified wood, fused shale and Gronlid siltstone. Exotic materials included Montana agate and Knife River flint (Zurburg 1991).

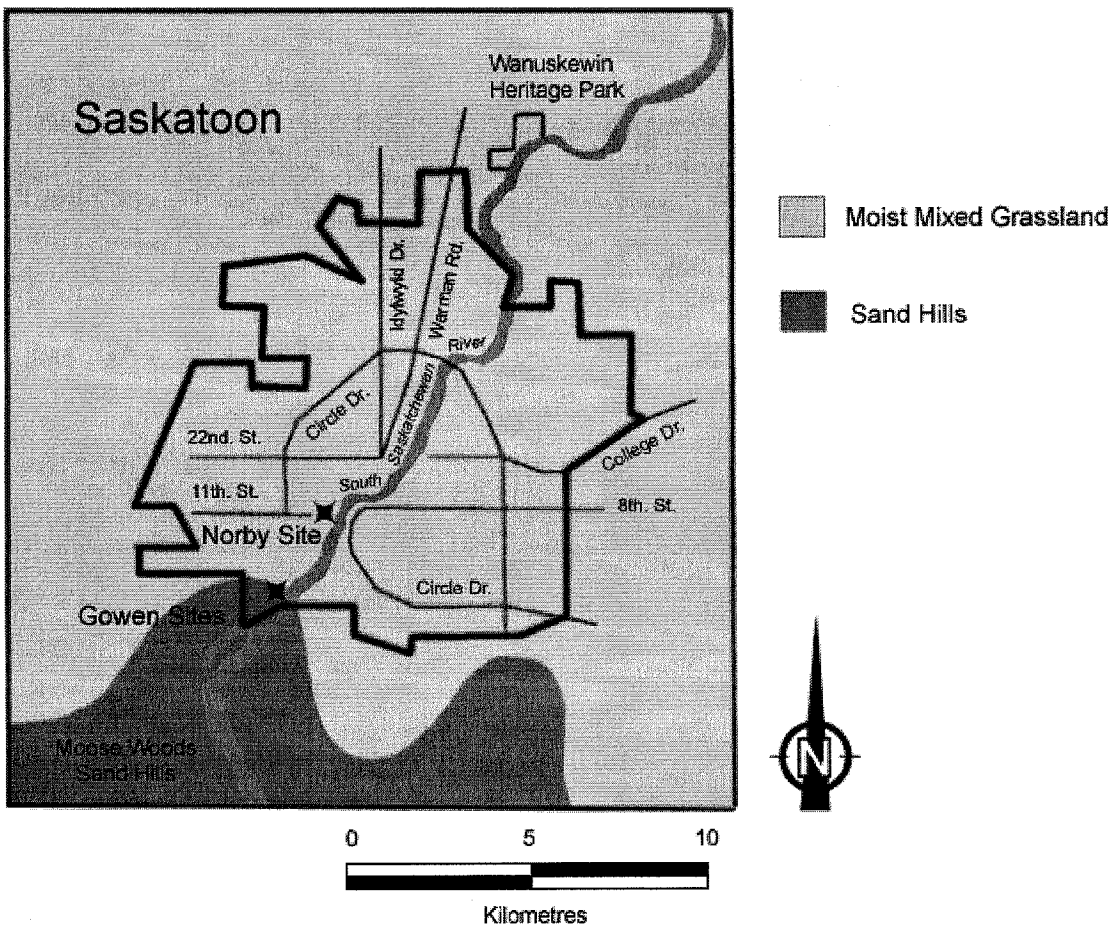
The Norby site faunal collection was found to be primarily composed of bison, but also included two rabbit metatarsals and the complete lower dentition from a large canid (Zurburg 1991). Bison bone recovered from the bone bed was highly fragmented, but constituted a total of 2,970 complete and fragmentary specimens. MNI values were relatively high with a count of 26 individuals as determined from fused central and fourth tarsals (Zurburg 1991). Zurburg (1991) describes the condition of bone at the Norby Site as having undergone a high degree of weathering. As a result of this, cut-mark and carnivore/rodent modification analyses were difficult to undertake (Zurburg 1991). Little evidence of bone breakage for the purposes of marrow extraction exists at the site and most long bones appear to have been deposited whole. Weathering was noted with some regularity on the upper surfaces of bones, suggesting that the assemblage was subjected to little post-depositional movement (Zurburg 1991).

Only 3.2 % of the Norby site bone was identifiable (Zurburg 1991). Sex determinations based on cranial elements were impossible to make due to this high degree of fragmentation. Bivariate plots of long bone, carpal and tarsal measurements seemed to indicate a predominance of male specimens (Zurburg 1991). Mandible samples were grouped and aged primarily on the basis of mandibular molar metaconid height measurements and dental wear patterns (Zurburg 1991). In addition, maxillary molar paracone height measurements were also taken (Zurburg 1991). The resultant mortality curve for the Norby site bison reflects the age range composition expected within a normal herd. Zurburg (1991) suggests that this profile reflects the catastrophic nature of the Norby site kill. A lack of immature specimens is a simple compositional reflection of a typical male herd (Zurburg 1991). Furthermore, age determinations

reflect the seasonally restricted nature of bison calving and set the kill event to sometime in January or February (Zurburg 1991).

Zurburg (1991) also suggests that the Norby bison represent a transitional form. The long bone measurements determined from the assemblage clustered around and below the range previously determined for *Bison bison antiquus* and above that of *Bison bison bison* (Zurburg 1991). Zurburg concludes that the Norby bison likely represent a population of the transitional species, *Bison bison occidentalis* (Zurburg 1991). Unfortunately, due to the urban constraints of excavation, much of the necessary geomorphological data required for a better understanding of procurement strategies at the site could not be collected (Zurburg 1991). As a result, the manner in which so many prehistoric bison were trapped, may never be known. This is unfortunate as Mummy Cave age sites are rare and provide comment on a time of extreme environmental change in the Northern plains. The Norby site materials are currently housed at the archaeological and paleoenvironmental laboratory located in the interpretive centre at Wanuskewin Heritage Park. All specimens were catalogued and manually cleaned by a lab technician at the University of Saskatchewan. The information was then entered into a Data Base III computer management system.

Figure A4. Archaeological Sites Within the City Limits of Saskatoon



A.5 Wanuskewin Heritage Park

A.5.1 Background

Wanuskewin Heritage Park is located approximately 3 km north of Saskatoon (Walker 1999). The area of the park is roughly one square mile, and it is currently bisected by Opimihaw Creek (formerly Tipperary Creek); a tributary of the South Saskatchewan river which forms the southern boundary of the park. The valley walls of Opimihaw Creek are characterized by coulee depressions, while the valley bottom is lined with terraces and point bars (Walker 1999). These features contain "a large number of archaeological sites spanning the past 6,000 years of local prehistory" (Walker 1999:2). Approximately 19 sites are located in the park, including various habitation areas, bison procurement sites, tipi rings and a boulder alignment feature (Walker 1999). The archaeological significance of the area has been understood since the 1930s and it has been the site of sustained investigation since. Dr. E. G. Walker of the Department of Anthropology and Archaeology at the University of Saskatchewan, initiated a detailed archaeological survey of the region in 1982. It was at this time that the majority of the sites within the lands that currently comprise the park were described. The majority of these sites are essentially undisturbed and well-suited to intensive, long-term study (Walker 1999).

Wanuskewin was officially designated as a National Historic Site in 1987. The interpretive park was opened in 1992 and includes an interpretive facility and interpretive trails as well as a program of on-going archaeological excavations and analysis. The interpretive centre also functions as a cultural centre for local aboriginal groups. Wanuskewin has been cited as a model of involvement and cooperation with the First Nations community (Walker 1999). Wanuskewin also houses an on-site archaeological and paleoenvironmental laboratory which is operated by the University of Saskatchewan. The laboratory coordinates and oversees all research in the park, but is also active in public education programming (Walker 1999:). For the last several years, the Department of Anthropology and Archaeology at the University of Saskatchewan has operated an archaeological field school in the park. Much of the archaeological material recovered from these excavations and others from earlier in the parks history, are currently stored at the laboratory facility in the park.

A.5.2 The Amisk Site (FbNp - 17)

The Amisk site represents a multi-component prehistoric campsite on a hill-wash slope in the Opimihaw Creek valley running through Wanuskewin Heritage Park. The site is situated in the valley roughly 450 meters north of where the creek empties into the river (Amundson 1986). Dr. Walker initially described the site in 1983 during his archaeological resource assessment of the park (Amundson 1986). There are seven cultural layers (levels) at the site separated by colluvial sediments. Level one may include as many as three separate occupations while the remaining six appear to be singular (Amundson 1986). Only levels one, four and five produced diagnostic materials. Excavation of the site was begun in May of 1984 (Amundson 1986). The 1984 field season concentrated on determining the vertical extent of the site and defining the cultural sequence. In 1985, the excavation was expanded and completed. In the end, a total area of 42 m^2 was opened and excavated at the Amisk site (Amundson 1986). All materials recovered from the excavation are currently housed at the University of Saskatchewan archaeological and paleoenvironmental laboratory in the Wanuskewin interpretive centre.

Only two identifiable cultural complexes are represented at the Amisk site. Roughly 15 complete and fragmented projectile points were recovered from the first level. These were determined to represent seven Plains Side-notched points, two Prairie Side-notched points and two Avonlea style points (Amundson 1986). The late side-notched series points recovered from level one suggested a compression of the stratigraphy. Amundson (1986) states that three radiocarbon assays that were obtained from bison bone collagen samples from the first level, may suggest that the level represents at least three distinct occupations (Amundson 1986). The other identified complex at Amisk is Oxbow. Diagnostic points were recovered from levels four and five (Amundson 1986). Of seven projectile points collected from level four, six were identifiable as Oxbow points (Amundson 1986). An un-calibrated radiocarbon date obtained from bone collagen assigned the level to an age of 4,015 ± 195 radiocarbon years. Only one basal point fragment was recovered from level five, however, it was identifiable as an Oxbow point (Amundson 1986). A radiocarbon assessment of bone

collagen dated this level to approximately 4,120 ± 190 radiocarbon years in age (Amundson 1986).

Level four occurs between about 55 and 80 cm below surface. Cultural materials occur within a 10 to 15 cm thick paleosol surrounded by a matrix of loamy sand (Amundson 1986). Artifacts were found to be densely scattered throughout the excavated area. Aside from projectile points, the lithic assemblage included a selection of tools and debitage. The recovery of several end and side scrapers, as well as bifacial knives and utilized flakes suggest that various processing activities were being undertaken. 96% of the recovered lithic artifacts were classified as debitage (flakes and shatter). The identification of several cores along with a significant amount of debitage, would seem to indicate that tool manufacture and repair were also prevalent activities. Locally available raw materials such as chert, jasper, Swan River chert and quartzites of varying grain size' dominate the assemblage. However, at least two types, chalcedony and a welded volcanic tuff, are considered to be exotic (Amundson 1986).

Preservation of bone in the fourth level was excellent (Amundson 1986). Species other than bison that were discovered in this level include 12 clam specimens, 10 canid specimens, three ground squirrel specimens and a single beaver ulna (*Castor canadensis*) (Amundson 1986). Bison from the level constituted a NISP of 8,090 representing a minimum of four individuals (Amundson 1986). Although no further analysis was undertaken, bison remains from this level are assumed to be representative of the modern species of plains bison (*Bison bison bison*). Several of the bones displayed cut marks and were scattered and fragmented indicating that butchering and some bone breaking activities had occurred in this occupation. Amundson (1986) suggests that this reflects a pattern of procurement in which animals were transported to Amisk from a kill site of unknown location (Amundson 1986).

A roughly circular hearth feature was also identified during excavation of level four. Soil from within the feature was described to be fire-reddened and blackened. It contained several pieces of charcoal and charred bone fragments. In addition, two of the projectile points, two cores and lithic debitage were also discovered within the vicinity of the feature. Amundson (1986) suggests that this hearth feature may well have been a central area around which several activities, including cooking, food processing and

possibly tool manufacture and repair, may have occurred. Thus, the Amisk site Oxbow components represent a campsite in which the processing of bison was a dominant activity. This assessment seems to agree with interpretations of Oxbow culture from other sites in southern Saskatchewan (Amundson 1986).

A.5.3 The Thundercloud Site (FbNp - 25)

The Thundercloud site was originally detailed in Walker's 1982 survey of the Wanuskewin Heritage Park lands. The site itself is situated on a point bar to the east side of Opimihaw creek in the northern reaches of the Park (Webster 1999). All artifacts recovered at the Thundercloud site are currently housed at facilities in the archaeological and paleoenvironmental laboratory located within the interpretive centre at Wanuskewin Heritage Park. Test pits "revealed the presence of a rich multi-component site with relatively intact stratigraphy" (Webster 1999:6). Full scale excavations began in the summer of 1993 as part of the Department of Anthropology and Archaeology field school offered through the University of Saskatchewan. Excavation continued for six field seasons and was completed in the summer of 1998 (Webster 1999). At least seven buried soil horizons (levels) containing ten occupations have been identified at the Thundercloud site (Webster 1999).

The first level represents a Late Prehistoric occupation and was found to contain a variety of projectile points including historic metal points, Prairie Side-notched, Plains Triangular and Plains Side-notched styles (Webster 1999). Levels 2 and 3 contain a variety of point types as well. These include Prairie Side-notched, Plains Triangular, Plains Side-notched, Avonlea and Besant styles (Webster 1999). Unfortunately, stratigraphic separation was difficult to discern for much of the first three levels and as a result, individual occupations were problematic to assess (Webster 1999). While level 4 is separated from level 3 by a thin sterile layer of gravelly mud, it is poorly developed and contains no diagnostic projectile points (Webster 1999).

Level five contains materials associated with the McKean Cultural Complex and included seven points that were identifiable as either Duncan, Hanna or McKean Lanceolate varieties (Webster 1999). This level may represent more than one occupation. One un-calibrated radiocarbon date was obtained from a bison humerus

from this level giving a date of 4040 ± 90 un-calibrated R.C.Y. BP (Webster 1999). Level six occurs either directly below level five or with a thin layer of sterile sand separating the two. Of the projectile points recovered from level six, only those identified as Oxbow points can be confidently attributed to the level (Webster 1999). Level seven is not continuous throughout the site and contains no diagnostic materials (Webster 1999).

Water screening and hand-flotation techniques were used on a variety of samples from the Thundercloud site. These investigations were designed in part to evaluate the effectiveness of such fine screening techniques (Webster 1999). As a result, all levels and primary features from the site possess fine screen enhanced assemblages. Artifacts recovered by such methods include micro-vertebrate remains, gastropod shells and a variety of seeds and carbonized organics (Webster 1999). The assemblage of specimens recovered through the fine screening techniques is too large to effectively describe in cursory detail.

While bison dominated the total recovered faunal assemblage, other specimens included pronghorn, elk, deer, various canids, mustelids, leporids and rodents along with a limited assemblage of birds, amphibians, reptiles and fish (Webster 1999). In addition, a selection of clams, bivalves and snails was also collected. While recovered faunal remains were often highly fragmented, the preservation of bone material was considered to be good. With the exception of level seven, all horizons at the Thundercloud site provided MNI counts indicating at least three or more individual bison. Seasonality was examined for all levels using aging techniques on immature bison teeth, when available, and otherwise using the total faunal assemblages as indicators (Webster 1999).

Lis Mack (1999) undertook an analysis of the Thundercloud lithic assemblage. Level five is considered to represent a compacted stratigraphic horizon consisting of a single level throughout the majority of the site with three separate occupations present in the western portion of the site (Mack 1999). All three are associated with the McKean Complex. Tools, other than projectile points, that were recovered through excavation from this level include four stone knives, two bifacial perforators, four end scrapers, three cores and 2,260 pieces of debitage (flakes and shatter). The most

common material types were of local origin, although some exotic materials, such as obsidian and Knife River Flint were also noted (Mack 1999).

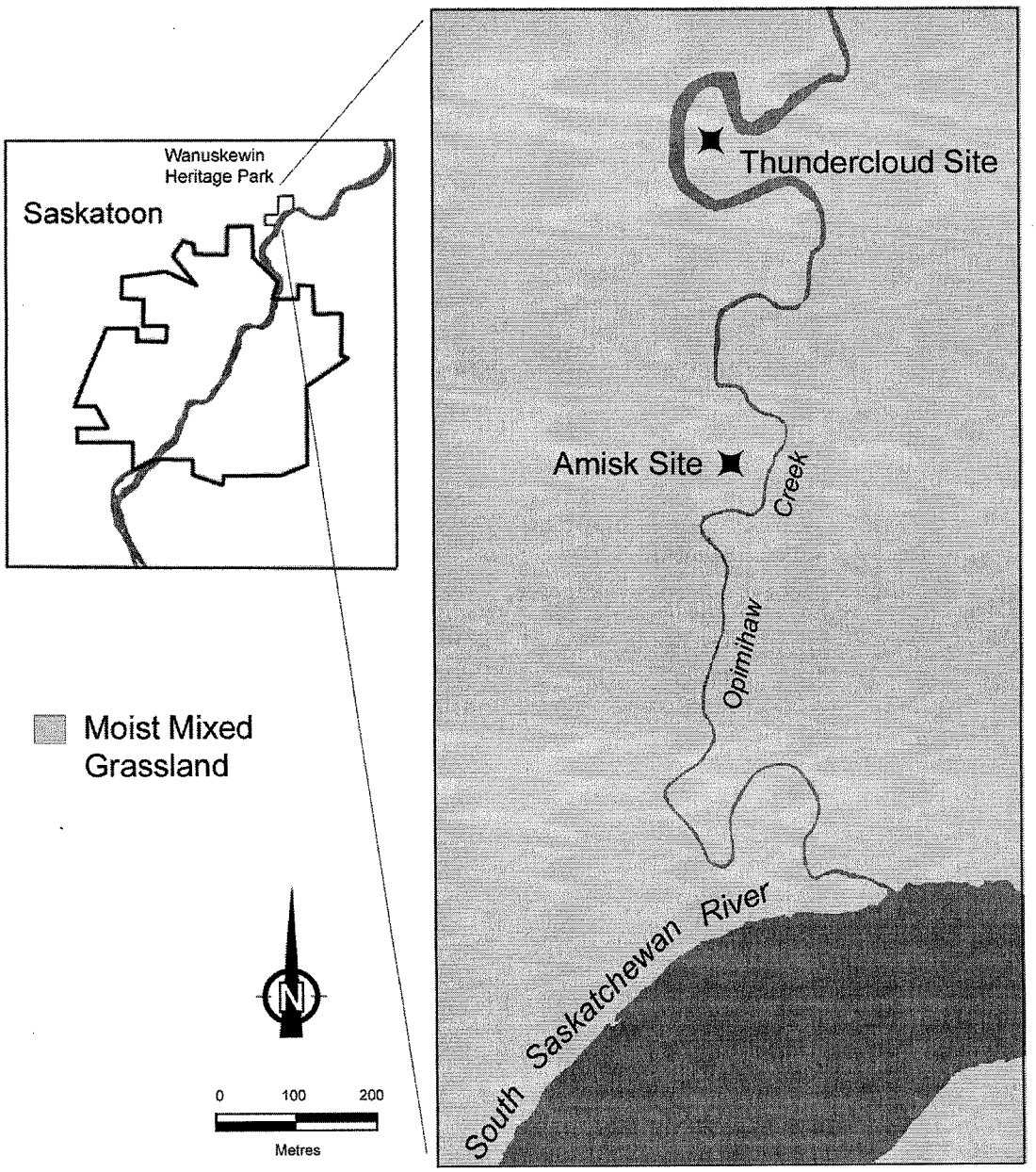
Of nine excavated features in level five, six were considered to be hearths, associated with large quantities of fire-broken rock. Two other features consisted of organic stains that may be related to the hearth features. A ninth feature consisted of a concentrated area of heavily processed, but otherwise un-modified bone. Concentrations of lithic debitage seem to surround the various hearth features potentially identifying these as areas in which tool sharpening occurred during work related activities (Mack 1999). Despite the fact that the majority of faunal remains in level five do not appear to have been burned or boiled, they do occur in proximity to the hearth features, suggesting that they may have been associated with the activities occurring in these areas. Fracture patterns amongst many of the long-bones may indicate that marrow extraction for immediate consumption was occurring (Webster 1999).

The faunal assemblage for level five, although comprised of 24 different taxa, is dominated by the remains of bison. A minimum of eight individuals is thought to be represented by the assemblage (Webster 1999). All are assumed to represent modern plains bison (*Bison bison bison*). Other recovered species constitute an interesting mix of medium and small sized mammals, along with specimens of various birds, amphibians and invertebrates (Webster 1999). Faunal diversity in level five was in fact, greater than that of any other level examined at the Thundercloud site. The analysis of five immature bison and the presence of other species which normally hibernate or migrate during winter led to the conclusion that during the time period represented by level five, the Thundercloud site had been occupied more than once. Dental wear and eruption sequences amongst the immature bison suggested at least two kill events; one occurring in the spring and the other during the winter (Webster 1999). This interpretation was easily reconciled with the previously mentioned stratigraphic interpretations of level five.

The frequency of various bison skeletal elements recovered during excavation of level five, suggest that only high utility items were being brought to the site (Webster 1999). This fact, combined with the identification of processing areas surrounding several hearths, lends support to the idea that the various level five occupations

represent the remains of a processing area/camp site. Although remains within the component would seem to suggest that the inhabitants of the site were primarily small-scale bison hunters, identification of the processed remains of other mammals, birds, amphibians and invertebrates would suggest that McKean subsistence strategies were somewhat more complex (Webster 1999). Inferences into the paleoenvironments represented in the Thundercloud cultural sequence were also generated through analogy with the various species represented and an understanding of their known ranges (Webster 1999). Webster (1999) suggests that evidence from the Thundercloud site may support the idea that "conditions 4,500 to 3,000 years ago could have been warmer and/or drier than those of today but not significantly cooler and/or wetter" (Webster 1999:217).

Figure A5. Archaeological Sites Located Within the Boundaries of Wanuskewin Heritage Park



Appendix B. RADIOCARBON DATES

B.1 Introduction

The interpretations generated in this thesis result from the application of new analytical techniques to previously excavated archaeological materials. Because this study required comparisons to be made of bison remains from different time periods, selection was limited to sites possessing materials from dateable contexts. A list of previously determined radiocarbon dates from each site and the associated cultural components analyzed in this study are presented in Table B1. The individual dates presented in this table are given in an un-calibrated but normalized form. Normalization involves a calculation to correct for the effects of isotopic discrimination based on the $^{13}C/^{12}C$ ratio of the radiocarbon sample. Unfortunately, all but the most recent radiocarbon dates do not usually provide $\delta^{13}C$ values. Dr. R. E. Morlan of the Canadian Museum of Civilization has compiled an online database of radiocarbon dates including those from Saskatchewan. This database can be accessed at the following web address:

http://www.canadianarchaeology.com/radiocarbon/card/card.htm#index

In most cases, this database provides normalization using an estimated $\delta^{13}C$ value. In some instances, however, $\delta^{13}C$ values were measured in the original study and were thus used to normalize these dates. The references from which these data derive are listed in the bottom row of the table. Those dates which are underlined have been discredited by the author(s) of the original source reference and are thus not utilized in any estimation of the site age. The estimated radiocarbon ages are a rough average of the available, usable dates from each site component. These estimates have been made from the normalized data.

Table B1. Previously Determined Un-calibrated Radiocarbon Dates

Site	Heron Eden	Norby	Gowen	Amisk	Harder	Thundercloud	Fitzgerald	Tschetter
Borden #	EeOi-11	FaNq-56	FaNq-32	FbNp-17	FbNs-1	FbNp-25	ElNp-8	FbNr-1
Cultural Association	Cody Complex (Eden)	Mummy Cave, Terminal Paleoindian	Mummy Cave (Gowen)	Oxbow	Oxbow	Mckean (Level 5)	Besant	Oldwoman's Complex (Prairie side-notched)
Previous Un-calibrated dates	8240 ± 200* (S-3208) 9000 ± 130* (S-3309) 9010± 120* (S-3114) 9290 ± 110* (S-3308) 10290 ± 100* (S-3118)	5640 ± 120* (S-3206) 5820 ± 110* (S-3205) 5965 ± 265* (S-3006)	5670 ± 110** (S-2037) 5990 ± 170* (S-2036) 6000± 130* (S-1970) 6160 ± 160* (S-1971)	4095 ± 195* (S-2536) Level 4 4200 ± 190* (S-2535) Level 5	Dyck 3400 ± 120* (S-490) 3505 ± 105* (S-668) Morlan 3570 ± 140 (S-3453) ($\delta^{13}C$ –15.5) 4335 ± 90 (S-3452) ($\delta^{13}C$ –15.9) 4410 ± 150 (S-3452) ($\delta^{13}C$ –18.3)	4145 ± 90 (S-3645) ($\delta^{13}C$ –18.4)	1240 ± 170* (S-3547) 1350 ± 140* (S-3546) 1420 ± 60* (Beta-69004) 1590 ± 90* (Beta-69005)	1000 ± 45* (S-1631) 1085± 75* (S-669) 1100 ± 100* (S-2225)
Approximate Radiocarbon Age Yrs. BP	9100	5800	6000	4150	3500 (4400)	4150	1350	1050 (1035 ± 40)
References	Corbeil 1995	Zurburg 1991	Walker 1992	Amundson 1986	Dyck 1977 Morlan 1994	Webster 1999	Hjermstad 1996	Linnamae 1988

*** Sample normalized using -20 as an estimated $\delta^{13}C$ value**
**** Sample normalized using -25 as an estimated $\delta^{13}C$ value**
Underlined dates have been previously discredited by the author(s) of the source reference

B.2 Rafter Radiocarbon Laboratory AMS Dates

Although the specimens analyzed in this study each derive from previously dated contexts, it was decided that a single sample from each represented time period would be re-dated to provide a verification of the chronological associations between the sites. There were several reasons why a reevaluation of the dates was desirable. A key consideration was the fact that many of the original site dates were determined decades prior to this study. There have been several advances in methods of sample preparation and date determination over the last 50 years (see Chisholm 1989 and Schoeninger and Schwarcz 1991 for review). Additionally, several different labs provided the various dates. It is difficult to know what procedural differences may have been employed at the various labs at different times, and what effect that may have had on the dates themselves. As well, the reporting of these dates is not always consistent. Although most are presented in a raw form, at least one has been normalized. Normalization of the other dates is only possible through use of an estimated $\delta^{13}C$ value.

The methodology associated with this thesis afforded the author the opportunity to address these concerns. Stable isotope analysis employs much of the same methodology that is required for radiocarbon dating. Dating bone collagen necessitates that the collagen from each specimen be chemically extracted and purified (see Appendix D). This preparation had already been performed for every sample in this project that was analyzed for stable isotope ratios. In addition, the analysis of hydrogen stable isotope ratios required that an "offline" combustion and gas extraction was also performed for each sample (see appendix D). A by product of this was the production and collection of purified CO_2 gas from each sample. Radiocarbon dating of bone collagen by accelerator mass spectrometry (AMS) requires the extraction of CO_2 from prospective samples. Thus, ampoules of purified CO_2 from every specimen analyzed in this study, were available for immediate AMS dating. Ultimately, the samples used to acquire the new AMS dates were subject to a greater degree of scrutiny and controlled preparation than were those from which the original dates were obtained.

A total of eight CO_2 samples representing each of the temporally distinct sites, were sent to the Rafter Radiocarbon Laboratory in New Zealand for AMS radiocarbon

dating. Each sample to be dated was randomly selected from amongst those specimens that produced good quality collagen (see Appendix E). Each of the CO_2 ampoules contained gas that had been collected from bone collagen samples that were prepared and combusted at the National Hydrology Research Centre Stable Isotope Laboratory in Saskatoon (see Appendix D). The resultant dates and all supplementary information are presented in Table B2. Calibrations were performed using the INTCAL98 database (see Stuiver *et al.* 1998). Radiocarbon age, $\delta^{14}C$, $\Delta^{14}C$ and percent modern are as defined by Stuiver and Polach (1977). The sample ID and lab ID of each specimen were designated by the author for the purposes of this thesis (see Appendix C) while the sample codes and lab codes were assigned by the Rafter Radiocarbon Lab.

For the most part, the new AMS dates verify the older conventional dates. This assessment is based on comparisons between the un-calibrated radiocarbon ages in each case. The majority of the new dates provide age estimates within about 100-200 years of the earlier dates. Although the dates initially reported by Dyck (1977) for the Harder site are several hundred years younger than the Rafter AMS date, Morlan's (1994) dates for the Harder site are much closer in age. Morlan's (1994) re-examination of the Harder site materials suggests reasons why the older dates are more acceptable (see Appendix A).

The Rafter AMS dates for the Thundercloud, Norby and Tschetter sites do differ from the originally reported radiometric dates. Specimens from the Thundercloud site have recently been re-dated independently of this project. New dates for the level five materials provide age estimates that are very close to the Rafter AMS date for this site (Webster 2002, Personal Communication). The Rafter AMS date for the Tschetter site was much too recent and obviously erroneous. A re-examination of the sample that was sent to the Rafter Radiocarbon laboratory, suggested that a specimen un-related to the Tschetter site had been accidentally included and processed with the Tschetter materials. Subsequently, the Rafter AMS date for the Tschetter site was discredited and the sample that provided the date was removed from further analysis in this study.

The Rafter AMS date from the Norby site proved to be more problematic. The difference between it and the mean age from the previous conventional dates was more than 1,000 radiocarbon years. Nevertheless, the Rafter AMS date still falls to within the

age range normally ascribed to Mummy Cave Complex occupations (see Appendix A). The recovery of a terminal Paleoindian period Manitoba style projectile point may suggest an earlier age for the Norby site cultural material. There is little evidence, however, to suggest that the Norby cultural component represents more than one occupation. Zurburg (1991) asserts that the Norby site itself is evidence for the later occurrence of the Manitoba style in the Canadian plains. An additional consideration concerns the apparent low collagen concentrations that are associated with the Norby site bone (see Appendix E). Norby bone samples required stringent sample preparation, both to remove contaminants and to appropriately scrutinize the collected collagenous residues. Such measures were applied to all of the samples prepared for this thesis, to the best ability of the author. There is unfortunately, no way to assess the procedures that were utilized for the radiometric Norby dates. For these reasons it is felt that the Rafter AMS date probably represents the best age estimate for the Norby specimens.

It was subsequently decided that the Rafter AMS dates provided the best age estimates for all of the sites analyzed in this thesis, with the exception of the Tschetter site. The age reported in this project for the Tschetter site represents an aggregate mean of the dates presented by Linnamae (1988). The reported dates associated with the cultural components from the other sites analyzed in this project are all based upon the Rafter AMS dates determined during this study. Generally, the author had more control over the preparation of the samples from which the Rafter dates were obtained. Additionally, the nature of the current study yields a wealth of independent information concerning the quality of submitted samples that is not normally available to researchers upon receiving the results of radiocarbon dating (see Appendix E). Together, all of these factors produce a high degree of confidence in the Rafter AMS dates.

Table B2. Rafter Radiocarbon Laboratory AMS Dates

Site	Heron Eden	Norby	Gowen	Amisk	Harder	Thundercloud	Fitzgerald	Tschetter
Borden #	EeOi-11	FaNq-56	FaNq-25/32	FbNp-17	FbNs-1	FbNp-25	ElNp-8	FbNr-1
Cultural Association	Cody Complex (Eden)	Mummy Cave, Terminal Paleoindian	Mummy Cave (Gowen)	Oxbow	Oxbow	McKean (Level 5)	Besant	Oldwoman's Complex (Prairie side-notched)
Sample ID	A2	C7	B5	D1	E12	F3	G2	H9
Lab ID	12451	12474	12466	12482	12495	12501	12503	12525
Sample Code	R 26941/1	R 26941/3	R 26941/2	R 26941/4	R 28001	R 26941/6	R 26941/7	R 26941/8
Lab Code	NZA 15745	NZA 15747	NZA 15746	NZA 15748	NZA 15776	NZA 15749	NZA 15750	NZA 15751
Measured $\delta^{13}C$	-18.8	-17.8	-17.5	-19.1	-17.8	-19.4	-18	-18.2
Radiocarbon Age Yrs. BP (Normalized)	9168 ± 50	7036 ± 45	5863 ± 55	4358 ± 45	4221 ± 45	3382 ± 55	1563 ± 45	-28 ± 55
Calibrated Range Yrs. BP (2 Sigma)	10485 - 10218	7949 - 7743	6795 - 6510	5043 - 4838	4856 - 4617	3817 - 3470	1543 - 1341	
Calibrated Range Yrs. BP (1 Sigma)	10397 - 10235	7931 - 7792	6740 - 6636	4970 - 4855	4839 - 4714	3689 - 3562	1522 - 1405	
$\delta^{14}C$	-678.6 ± 2	-580 ± 2.5	-513.6 ± 3.2	-415.4 ± 3.4	-403.7 ± 3.5	-340.2 ± 4.4	-170.3 ± 4.8	11.2 ± 7.2
$\Delta^{14}C$	-682.6 ± 2	-586.1 ± 2.4	-521.1 ± 3.2	-422.4 ± 3.4	-412.5 ± 3.4	-347.8 ± 4.3	-182 ± 4.7	-2.8 ± 7.1
% modern	31.74 ± 0.2	41.39 ± 0.24	47.89 ± 0.32	57.76 ± 0.34	58.75 ± 0.34	65.22 ± 0.43	81.8 ± 0.47	99.72 ± 0.71
Date	Aug 26, 2002	Aug 26, 2002	Aug 26, 2002	Aug 26, 2002	Sept 02, 2002	Aug 26, 2002	Aug 26, 2002	Aug 26, 2002

Appendix C. RAW CATALOG DATA

C.1 Introduction

Once the archaeological sites to be examined in this study had been researched and selected, the specific faunal elements that were to be analyzed from each site were acquired from storage. A record of each bone was made. Because isotopic analyses result in the modification or destruction of analyzed materials, information recorded by the researcher prior to the analysis may be the only source of data concerning the original sample to survive. In this case, these records included the following catalog data concerning the physical attributes of each specimen and a series of photographs that reside in the possession of the author. The following catalog presents a brief qualitative and metric analysis of each faunal element from which samples to be analyzed in this thesis were obtained.

Each specimen was assigned an alphanumeric designation by which it was subsequently identified throughout this project. The designations I and J are used to identify the locations from which modern samples were obtained. Several dietary samples were collected in conjunction with the modern bone specimens. These are separately recorded at the end of the catalog. Although each of the dietary specimens has been assigned a sample ID, no other information was recorded. Because all of the prehistoric materials were originally recovered during previous excavations, some original catalog data from these projects may be relevant to the identification of specific samples. These data have been recorded in the "comments" section of this catalog where relevant.

The catalog system presented here is based upon the faunal catalog used for the University of Calgary Field School during the excavation of the Tuscany Archaeological Project in Northwest Calgary. It is used with the permission of the

project director Dr. Gerald Oetelaar of the Department of Archaeology, University of Calgary. This catalog system was chosen because of the author's familiarity with it. Hopefully, this familiarity will provide a degree of consistency in the catalog. The goal was to record information for posterity rather than for the purpose of contributing directly to an analysis. As a result, a laborious attempt at researching and constructing a new catalog system was not warranted. A key to the catalog is presented first, followed by the catalog data presented in tables.

C.2 Catalog Key

1. **SAMPLE IDENTIFICATION**: a two digit alphanumeric code used to designate the site from which the specimen originated (See Codes Below) and the site specific specimen number *(eg.* A1*)*.

   Codes:

   A - Heron Eden Site (EeOi-11)
   B - Gowen Site B (FaNq-32)
   C - Norby Site (FaNq-56)
   D - Amisk Site (FbNp-17)
   E - Harder Site (FbNs-1)
   F - Thundercloud Site (FbNp-25)
   G - Fitzgerald Site (ElNp-8)
   H - Tschetter Site (FbNr-1)
   I - Wichita Mountains Wildlife Refuge, Oklahoma, USA.
   J - Bison Processer, Saskatchwan (Yorkton and Saskatoon)

2. **SITE**: the Borden designation of the archaeological site from which the specimen was recovered (*eg.* EgPn-377) (If applicable).

3. **SPECIES**: the name of the species identified:
   animal - only if cannot determine whether mammal, bird or fish
   mammal - not bird or fish but can't determine exact species
   cf. Genus - can determine *Genus* but not the *species (eg. cf. Bison)*
   Genus species - can determine exact species (eg. *B. bison*)

4. **ELEMENT**: an abbreviation of the element as identified using the codes provided below:

As - astragalus
At - atlas
Ax - axis
Bf - bone fragment
Ca - calcaneum
CaVe - caudal vertebra
CeVe - cervical vertebra
CP - cuneiform pes
Cu – cuneiform
Cr – cranium
Ep - indeterminate Epiphysis
Fe - femur
Hu - humerus
Il - ilium
Ir – incisors
Is - ischium
LBf - long bone fragment
Lm - lateral malleolus
Lu - lunate
LuVe - lumbar vertebra
Mc - metacarpal
Mg - magnum
Mn - mandible
Mo - molars/premolars
Mp - metapodial
Mt - metatarsal
Mx - maxilla
NC - naviculo-cuboid
OsCo - os coxa
Pa - patella
Pe - petrous
Ph I - first phalanx
Ph II - second phalanx
Ph III - third phalanx
Pi - pisiform
Pu - pubis
Ra - radius
Ri - rib
Sa - sacrum
Sc - scapula
Se - sesamoid
Sk - complete skeleton
Sp - scaphoid
St - sternum
Ul - ulna
Un - unciform
Te - tooth fragment
ThVe - thoracic vertebra
Ti - tibia

5. **PART**: part of the element represented.

0 INDETERMINATE
1 COMPLETE
2 PROXIMAL
3 MEDIAL
4 DISTAL
5 ANTERIOR
6 POSTERIOR

6. **FUSION**: the status of the epiphyseal fusion.

0 INDETERMINATE
1 UNFUSED
2 PARTLY FUSED
3 FUSION LINE
4 FUSED

7. **SIDE**: the side of the animal represented by the element.

0 INDETERMINATE
1 AXIAL
2 RIGHT
3 LEFT

8. **BURNING**: the extent of burning (*burning* defines discoloration all the way through whereas *staining* occurs only on the outside edges).

0 UNBURNED
1 BURNED BROWN
2 BURNED BLACK
3 BURNED GREY (blue grey)
4 BURNED WHITE (almost ash)
5 BLEACHED (can see bone structure)

9. **HUMAN MODIFICATION**: identify the nature of the modification to the bone *if there is any.*

0 INDETERMINATE
1 POLISHED
2 GROUND
3 WHITTLED
4 FLAKING
\ NO MODIFICATION

10. **TOOL TYPE**: identify the type of tool represented by the bone piece *if applicable.*

0 INDETERMINATE
1 FLESHER
2 AWL
3 FABRICATOR
4 HUSKER
\ NOT A TOOL

11. **BUTCHERING MARKS**: identify the type of butchering marks on the bone *If there are any*

0 INDETERMINATE
1 IMPACT SCAR
2 CUT MARKS (deep, V-shaped, usually parallel series)
3 HACK MARKS (smooth on one edge, broken on other)
\ NO MARKS PRESENT

12. **GNAW MARKS**: identify the type of gnaw marks present *if there are any*

0 INDETERMINATE
1 RODENT
2 CARNIVORE
3 HERBIVORE
4 RODENT CLAW MARKS
\ NO MARKS PRESENT

13. **PATHOLOGY**: note the presence or absence of pathologies for <u>all</u> bone (any abnormal bone growth eg. vertebrae, ribs and long bone joints usually affected).

0 ABSENT
1 PRESENT

14. **ABRASION**: note the presence or absence of root etching by plant roots (very common).

0 ABSENT
1 PRESENT

15. **WEATHERING**: identify the stage of weathering as determined by Behrensmeyer.

0 STAGE 0 (no sign of cracking or flaking; shiny surface still visible)

1 STAGE 1 (longitudinal cracks on shafts and mosaic cracking on facets)
2 STAGE 2 (outer bone layer has started to flake or curl)
3 STAGE 3 (lacks concentrically layered bone and shows rough fibrous texture)
4 STAGE 4 (bone is coarsely fibrous with cracks extending deep into bone)
5 STAGE 5 (bone is falling apart)

16. **PRESERVATION**: note the state of bone preservation after burial in sediments ("strength" is in relation to how much collagen is left in bone; how structurally sound is it? how stable is it?) *eg. "good" (3) = bone would break with a little pressure.*

0 INDETERMINATE
1 POOR
2 FAIR
3 GOOD
4 VERY GOOD
5 EXCELLENT

17. **COMPLETENESS**: assess how complete the bone fragment is.

0 INDETERMINATE
1 COMPLETE
2 75% OR MORE
3 50% OR MORE
4 25% OR MORE
5 LESS THAN 25%

18. **LENGTH**: a three digit number *(eg. 039)* for length measured in millimeters. Determine the maximum length to the nearest millimeter (largest dimension). If more than one piece present, take measurements on the largest piece.

19. **WIDTH**: a three digit number *(eg. 012)* for width measured in millimeters. Determine the maximum width to the nearest millimeter (largest dimension perpendicular to length). If more than one piece present, take measurements on the largest piece.

20. **THICKNESS**: a three digit number *(eg. 002)* for thickness measured in millimeters. Determine the maximum thickness to the nearest millimeter (dimension perpendicular to length and 90 degrees from width). If more than one piece present, take measurements on the largest piece.

21. **WEIGHT**: a four digit number *(eg. 031.2)* for weight measured in grams.

Determine the weight to the nearest tenth of a gram (be sure bone is dry). If more than one piece is present, take weight of all pieces.

22. **NUMBER OF PIECES**: note number of bone fragments. A two digit number (*eg. 05)* if less than 50 fragments; otherwise >50.

23. **COMMENTS**: note any additional comments (*eg.* original/site specific catalog information, taphonomic notes, etc.)

Table C1. Un-processed Sample Catalog.

Sample ID	Site	Species	Element	Part	Fusion	Side	Burning	Human Modification	Tool Type	Butchering	Gnaw Marks	Pathology	Abrasion	Weathering	Preservation	Completeness	Length (mm)	Width (mm)	Thickness (mm)	Weight (g)	Number of Pieces	Comments
A1	EeOi-11	Bison	Mc	1	4	2	0	\	\	\	\	0	0	3	4	1	199	068	041	263.4	1	Cat # EeOi-11 - 1050
A2	EeOi-11	Bison	Mc	1	4	2	0	\	\	\	\	0	1	3	4	1	187	062	037	235.9	1	Cat # EeOi-11 - 62
A3	EeOi-11	Bison	Mc	1	4	2	0	\	\	\	\	0	1	3	4	1	208	084	048	409.1	1	Cat # EeOi-11 - 8881
A4	EeOi-11	Bison	Mc	1	4	2	0	\	\	\	\	0	1	2	4	1	218	089	045	433.4	1	Cat # EeOi-11 - 946
A5	EeOi-11	Bison	Mc	1	4	2	0	\	\	\	\	0	0	3	4	1	201	079	045	374.0	1	Cat # EeOi-11 - 7845
A6	EeOi-11	Bison	Mc	1	4	2	0	\	\	\	\	0	1	2	4	1	216	079	045	388.7	1	Cat # EeOi-11 - 3165
A7	EeOi-11	Bison	Mc	1	4	2	0	\	\	\	\	0	1	3	4	1	217	065	039	272.6	1	Cat # EeOi-11 - 5718
A8	EeOi-11	Bison	Mc	4	4	2	0	\	\	\	\	0	1	3	4	3	206	065	035	195.6	1	Cat # EeOi-11 - 6215
A9	EeOi-11	Bison	Mc	4	4	2	0	\	\	\	\	0	1	3	4	2	091	085	040	351.9	1	Cat # EeOi-11 - 3559
A10	EeOi-11	Bison	Mc	1	4	2	0	\	\	\	\	0	1	4	3	1	213	081	042	454.6	3	Cat # EeOi-11 - 6714
A11	EeOi-11	Bison	Mc	1	4	2	0	\	\	\	\	0	1	3	4	2	219	081	049	376.4	4	Cat # EeOi-11 - 4885
A12	EeOi-11	Bison	Mc	1	4	2	0	\	\	\	\	0	1	3	4	2	123	081	040	379.7	15	Cat # EeOi-11 - 9071
B1	FaNq-32	Bison	As	1	4	2	0	\	\	\	\	0	1	3	3	1	079	062	055	107.7	1	Cat # FaNq-32 - 32 - 10, CaCO3 Buildup?
B2	FaNq-32	Bison	As	1	4	2	0	\	\	\	\	0	1	2	4	1	073	045	035	064.1	1	Cat # FaNq-32 - 2 - 81
B3	FaNq-32	Bison	As	1	4	2	0	\	\	\	\	0	1	3	3	1	075	048	034	070.0	1	Cat # FaNq-32 - 4 - 29
B4	FaNq-32	Bison	As	4	4	2	0	\	\	\	\	0	1	2	4	2	062	059	041	069.7	1	Cat # FaNq-32 - 33 - 127
B5	FaNq-32	Bison	As	2	4	2	0	\	\	\	\	0	1	3	3	5	057	042	023	015.9	1	Cat # FaNq-32 - 46 - 64, Proximal Lateral Trochlea
B6	FaNq-32	Bison	As	1	4	2	0	\	\	\	\	0	1	2	4	1	070	040	039	073.5	2	Cat # FaNq-32 - 43 - 48
C1	FaNq-56	Bison	Mc	1	4	3	0	\	\	\	\	0	1	2	4	1	209	084	045	398.9	2	Cat # 10b - 17

Sample ID	Site	Species	Element	Part	Fusion	Side	Burning	Human Modification	Tool Type	Butchering	Gnaw Marks	Pathology	Abrasion	Weathering	Preservation	Completeness	Length (mm)	Width (mm)	Thickness (mm)	Weight (g)	Number of Pieces	Comments
C2	FaNq-56	Bison	Mc	1	4	3	0	\	\	\	\	0	1	3	4	1	205	076	047	300.9	3	Cat # 10b - 21
C3	FaNq-56	Bison	Mc	1	4	3	0	\	\	\	\	0	1	2	4	1	093	077	041	305.6	6	Cat # 13a - 72
C4	FaNq-56	Bison	Mc	1	4	3	0	\	\	\	\	0	1	3	4	1	215	078	039	239.2	3	Unit 1, Exp. 3 # 9
C5	FaNq-56	Bison	Mc	1	4	3	0	\	\	\	\	0	1	2	4	2	112	079	044	276.1	9	Cat # 11a - 19
C6	FaNq-56	Bison	Mc	1	4	3	0	\	\	\	\	0	1	3	4	1	128	069	041	237.8	5	Cat # 15a - 1
C7	FaNq-56	Bison	Mc	1	4	3	0	\	\	\	\	0	1	3	4	2	130	057	040	292.0	9	Cat # 2d - 32
C8	FaNq-56	Bison	Mc	1	4	3	0	\	\	\	\	0	1	3	4	2	085	080	042	262.6	8	Cat # 9a - 29
C9	FaNq-56	Bison	Mc	1	4	3	0	\	\	\	\	0	1	3	4	2	060	044	041	175.8	14	Cat # 6a - 29
C10	FaNq-56	Bison	Mc	1	4	3	0	\	\	\	\	0	1	3	4	3	090	044	033	201.6	12	Cat # 25b - 23
C11	FaNq-56	Bison	Mc	1	4	3	0	\	\	\	\	0	1	2	4	1	221	086	043	378.8	7	Cat # 25b - 21
C12	FaNq-56	Bison	Mc	4	4	3	0	\	\	\	\	0	1	2	4	3	091	083	041	221.5	12	Cat # 9b - 34
C13	FaNq-56	Bison	Mc	4	4	3	0	\	\	\	\	0	1	2	4	2	092	080	041	270.5	14	Cat # 15b - 50
C14	FaNq-56	Bison	Mc	2	4	3	0	\	\	\	\	0	1	2	4	4	067	069	040	065.1	2	Cat # 21b - 46
D1	FbNp-17	Bison	Sp	1	4	2	0	\	\	\	\	0	1	1	4	1	047	041	023	011.3	1	Cat # FbNp-17 - 1029
D2	FbNp-17	Bison	Sp	1	4	2	0	\	\	\	\	0	1	1	4	1	049	043	025	013.1	1	Cat # FbNp-17 - 1689
E1	FbNs-1	Bison	Ti	4	4	3	0	\	\	\	\	0	1	1	4	4	191	067	047	139.8	1	Fn-982, Box 5 of 10, Bag 15s/0w, Distal Tibia 2of2
E2	FbNs-1	Bison	Ti	4	4	3	0	\	\	\	\	0	1	2	4	5	110	039	028	063.7	4	Fn-986, Box 9 of 10, Bag 42e/5s, July 15, Distal Tibia 1of2, 4 fragments re-fitted with glue, separated during cleaning
E3	FbNs-1	Bison	Ti	4	4	3	0	\	\	\	\	0	1	2	4	5	075	069	044	062.8	1	Fn-986, Box 9 of 10, Bag 42e/5s, July 15, Distal Tibia 2of2

Sample ID	Site	Species	Element	Part	Fusion	Side	Burning	Human Modification	Tool Type	Butchering	Gnaw Marks	Pathology	Abrasion	Weathering	Preservation	Completeness	Length (mm)	Width (mm)	Thickness (mm)	Weight (g)	Number of Pieces	Comments
E4	FbNs-1	Bison	Ti	4	4	3	0	\	\	\	\	0	1	3	4	5	075	073	044	060.8	1	Fn-982, Box 5 of 10, Bag 40s/5w
E5	FbNs-1	Bison	Ti	4	4	3	0	\	\	\	\	0	1	3	4	5	074	061	046	066.2	1	Fn-981, Box 4 of 10, Bag 20s/0w, Bag 1, Distal Tibia 1of2
E6	FbNs-1	Bison	Ti	4	4	3	0	\	\	\	\	0	1	2	4	5	077	060	058	058.2	1	Fn-981, Box 4 of 10, Bag 20s/0w, Bag 2, Distal Tibia 2of2
E7	FbNs-1	Bison	Ti	4	4	3	0	\	\	\	\	0	1	2	4	5	103	062	022	047.7	1	Fn-980, Box 3 of 10, Bag, 5+10n/0w, Distal Tibia 1of3
E8	FbNs-1	Bison	Ti	4	4	3	0	\	\	\	\	0	1	2	4	5	106	063	023	042.6	1	Fn-980, Box 3 of 10, Bag, 5+10n/0w, Distal Tibia 2of3
E9	FbNs-1	Bison	Ti	4	4	3	0	\	\	\	\	0	1	1	4	5	070	061	054	061.9	3	Fn-985, Box 8 of 10, Bag 42e/5+10n, Re-fitted glued 3 pieces, separated during cleaning
E10	FbNs-1	Bison	Ti	4	4	3	0	\	\	\	\	0	1	2	4	5	100	067	035	036.9	1	Fn-982, Box 5 of 10, Bag 15e/45s
E11	FbNs-1	Bison	Ti	4	4	3	0	\	\	\	\	0	1	2	4	5	068	060	029	029.0	1	Fn-982, Box 5 of 10, Bag 45s/0w, Distal Tibia 1of2
E12	FbNs-1	Bison	Ti	4	4	3	0	\	\	\	\	0	1	2	4	5	065	065	022	030.9	1	Fn-982, Box 5 of 10, Bag 45s/0w, Distal Tibia 2of2
E13	FbNs-1	Bison	Ti	4	4	3	0	\	\	\	\	0	1	2	4	5	061	041	031	014.8	1	Fn-984, Box 7 of 10, Bag 105s/0w, Sept. 14

Sample ID	Site	Species	Element	Part	Fusion	Side	Burning	Human Modification	Tool Type	Butchering	Gnaw Marks	Pathology	Abrasion	Weathering	Preservation	Completeness	Length (mm)	Width (mm)	Thickness (mm)	Weight (g)	Number of Pieces	Comments
E14	FbNs-1	Bison	Ti	4	4	3	0	\	\	\	\	0	1	2	4	5	060	060	042	028.0	1	Fn-981, Box 4 of 10, Bag 5s/0w, CaCO3 Buildup on interior cancellous tissue
E15	FbNs-1	Bison	Ti	4	4	3	0	\	\	\	\	0	1	2	4	5	047	045	037	047.9	4	Fn-986, Box 9 of 10, Bag 42e/5+10s, July 16, Re-fitted with glue, separated during cleaning into 4 pieces
F1	FbNp-25	Bison	Mt	4	4	2	0	\	\	\	\	0	1	2	4	3	178	070	037	175.9	1	Quad:NW, Unit:18s4e, Level C5, Bag 1, Location 10s/4e, Depth 47, Cat# 149
F2	FbNp-25	Bison	Mt	4	4	2	0	\	\	\	\	0	1	2	4	4	119	059	035	102.7	1	Quad:SE, Unit:20s4e, Level C5, Bag 25, Location 50s/82e, Depth 37, Cat# 186
F3	FbNp-25	Bison	Mt	4	4	2	0	\	\	\	\	0	1	1	4	5	069	036	033	057.0	3	Quad:NW, Unit:19s/1e, Level C5, Bag 91, Location 21s/26e, Depth 44, Cat# 379
G1	ElNp-8	Bison	Mt	1	4	3	0	\	\	\	\	0	1	2	4	1	244	065	049	373.1	1	Cat # ElNp-8 - 5727
G2	ElNp-8	Bison	Mt	1	4	3	0	\	\	\	\	0	1	2	4	1	240	057	049	284.5	1	Cat # ElNp-8 - 7696
G3	ElNp-8	Bison	Mt	1	4	3	0	\	\	\	\	0	1	2	4	1	238	058	048	253.0	1	Cat # ElNp-8 - 1666
G4	ElNp-8	Bison	Mt	1	4	3	0	\	\	\	\	0	1	2	4	1	252	072	059	381.4	1	Cat # ElNp-8 - 13938
G5	ElNp-8	Bison	Mt	1	3	3	0	\	\	\	\	0	1	3	4	1	245	067	056	328.4	1	Cat # ElNp-8 - 8661
G6	ElNp-8	Bison	Mt	1	4	3	0	\	\	\	\	0	1	3	4	1	262	068	054	403.7	1	Cat # ElNp-8 - 12103

Sample ID	Site	Species	Element	Part	Fusion	Side	Burning	Human Modification	Tool Type	Butchering	Gnaw Marks	Pathology	Abrasion	Weathering	Preservation	Completeness	Length (mm)	Width (mm)	Thickness (mm)	Weight (g)	Number of Pieces	Comments
G7	ElNp-8	Bison	Mt	1	4	3	0	\	\	\	\	0	1	2	4	1	238	059	047	262.3	1	Cat # ElNp-8 - 10910
G8	ElNp-8	Bison	Mt	1	4	3	0	\	\	\	\	0	1	2	4	1	257	067	056	332.1	1	Cat # ElNp-8 - 2173
G9	ElNp-8	Bison	Mt	1	3	3	0	\	\	\	\	0	1	3	4	1	245	067	052	391.2	1	Cat # ElNp-8 - 9372
G10	ElNp-8	Bison	Mt	1	4	3	0	\	\	\	\	0	1	2	4	1	230	059	049	233.4	1	Cat # ElNp-8 - 2647
G11	ElNp-8	Bison	Mt	1	4	3	0	\	\	\	\	0	1	2	4	1	241	065	058	368.0	1	Cat # ElNp-8 - 3762
G12	ElNp-8	Bison	Mt	4	4	3	0	\	\	\	\	0	1	2	4	3	152	070	039	209.5	1	Cat # ElNp-8 - 12103
G13	ElNp-8	Bison	Mt	4	4	3	0	\	\	\	\	0	1	2	4	3	135	068	038	174.4	1	Cat # ElNp-8 - 3958
G14	ElNp-8	Bison	Mt	4	4	3	0	\	\	\	\	0	1	2	4	4	115	066	037	129.9	1	Cat # ElNp-8 - 7962
G15	ElNp-8	Bison	Mt	4	4	3	0	\	\	\	\	0	1	2	4	2	164	060	035	155.6	1	Cat # ElNp-8 - 8017
H1	FbNr-1	Bison	Mt	1	4	3	0	\	\	\	\	0	0	4	3	1	261	066	062	434.6	1	Cat # FbNr-1 - 50 -76
H2	FbNr-1	Bison	Mt	1	3	3	0	\	\	\	\	0	0	4	3	1	256	062	057	381.2	1	Cat # FbNr-1 - 37 -128
H3	FbNr-1	Bison	Mt	4	3	3	0	\	\	\	\	0	0	4	3	3	187	070	044	276.8	1	Cat # FbNr-1 - 42 - 54
H4	FbNr-1	Bison	Mt	4	4	3	0	\	\	\	\	0	0	4	3	3	146	071	041	188.2	1	Cat # FbNr-1 - 68 - 63
H5	FbNr-1	Bison	Mt	4	4	3	0	\	\	\	\	0	0	3	3	4	129	072	036	180.7	1	Cat # FbNr-1 - 76 - 53
H6	FbNr-1	Bison	Mt	4	3	3	0	\	\	\	\	0	0	4	3	4	129	068	038	168.4	1	Cat # FbNr-1 - 68 - 65
H7	FbNr-1	Bison	Mt	4	3	3	0	\	\	\	\	0	0	3	3	4	140	060	034	128.2	2	Cat # FbNr-1 - 42 - 86, Separated during cleaning into 2 pieces
H8	FbNr-1	Bison	Mt	4	3	3	0	\	\	\	\	0	0	4	3	4	135	069	034	120.5	3	Cat # FbNr-1 - 37 - 11, Separated during cleaning into 3 pieces
H9	FbNr-1	Bison	Mt	4	3	3	0	\	\	\	\	0	0	3	3	4	094	061	034	095.6	1	Cat # FbNr-1 - 71 - 1
H10	FbNr-1	Bison	Mt	4	4	3	0	\	\	\	\	0	0	3	3	4	095	069	041	107.0	1	Cat # FbNr-1 - 25 - 237
H11	FbNr-1	Bison	Mt	4	3	3	0	\	\	\	\	0	0	3	3	4	094	056	034	065.9	2	Cat # FbNr-1 - 31 - 67, Separated during cleaning into 2 pieces

Sample ID	Site	Species	Element	Part	Fusion	Side	Burning	Human Modification	Tool Type	Butchering	Gnaw Marks	Pathology	Abrasion	Weathering	Preservation	Completeness	Length (mm)	Width (mm)	Thickness (mm)	Weight (g)	Number of Pieces	Comments
I1	N/A	Bison	Ph I	1	4	2	0	\	\	\	\	0	0	1	5	1	070	044	040	060.5	1	7 year old Female
I2	N/A	Bison	PH I	1	4	2	0	\	\	\	\	0	0	1	5	1	067	032	036	025.6	1	3.5 year old Male
I3	N/A	Bison	Sp	1	4	3	0	\	\	\	\	0	0	1	5	1	059	050	034	039.3	1	12 yr old Male
J1	N/A	Bison	Ra	1	4	2	0	\	\	\	\	0	0	0	5	1	186	070	040	395.3	3	2 year old Male (24 mos.)
J4	N/A	Bison	Sc	0	0	0	0	\	\	\	\	0	0	0	5	5					3	2 year old Male (24 mos.)
J5	N/A	Bison	Ra	1	2	3	0	\	\	\	\	0	0	1	5	1	282	088	062	380.2	1	18 mos. Male (Distal Ulna Present)
J6	N/A	Bison	Ri	1	0	3	0	\	\	\	\	0	0	0	5	1	528	033	024	225.9	1	1 year old Female (12 mos.)
Dietary Samples																						
I4																						Stomach Contents (Oklahoma)
J2																						Feed Pellets (Saskatoon, Wanuskewan, 2001)
J3																						Grass (Saskatoon, Wanuskewan, 2001)
J7																						Grass (Saskatoon, Wanuskewan, 2002)
J8																						Feed Supplement (Saskatoon, Wanuskewan, 2002)
J9																						Stomach Contents (Yorkton, 2002)
J10																						Grass (Yorkton, 2002)

Appendix D. DETAILED METHODOLOGY

D.1 Introduction

Archaeological samples to undergo stable isotope analysis usually require pre-treatment and preparation prior to the determination of isotopic ratios. These preparation techniques vary with different substances and are also dependent upon whether a project is concerned with the analysis of organically or inorganically bound elemental isotopes. This thesis involves the isotopic analysis of bone collagen, which is the primary organic component of bone. As such, each potential bone sample was subjected to pre-treatments that targeted the in-organic portion of the bone and allowed for extraction and preservation of the organic fraction. Contamination is also a potential concern when dealing with archaeological specimens. Taphonomic and diagenetic processes have the potential to alter skeletal elements mechanically and chemically post-mortem. Thus, most pre-treatments involve additional procedures that act to address contamination issues. There are at least a few different collagen extraction and purification methodologies that have been widely applied in archaeologically based isotope studies. Each presents advantages and disadvantages (See Moore 1989, Boutton 1991a and Schoeninger and Schwarcz 1991).

After extraction and purification the resulting collagen is ready to be analyzed. Because of instrumental requirements, substances to be analyzed must be converted into a gaseous state for stable isotope ratio measurements to be made. This is achieved by combusting the sample and then isolating and purifying the resultant gases. Isolation and purification allows for the targeting of various isotopes of any specific element (see Boutton 1991a). The means required to achieve combustion, isolation and purification will vary dependant upon which elemental isotopes are to be measured and the available instrumentation. Measurements of carbon and nitrogen isotope ratios are now largely automated, whereas determinations of hydrogen isotope ratios tend to require a more laborious methodology. All measured isotope ratios require standardization and

correction. Accurate determinations of hydrogen isotope ratios require the addition of equilibration procedures to account for the presence of labile hydrogen in organic samples (see Wassenaar and Hobson 2000).

D.2 Collagen Extraction

The collagen extraction and purification techniques used in this thesis derive from procedures described by Sealy (1986). Although other extraction methods have been described (see Stafford *et al.* 1987, DeNiro and Weiner 1988 and Schoeninger and Schwarcz 1991, for review) this technique provided certain advantages for the present study. One of the key benefits of using this procedure is its relative simplicity. Furthermore, it requires relatively minimal equipment and resources (Schoeninger and Schwarcz 1991). One drawback is that the correct application of this technique does require some previous experience because certain decisions regarding the readiness and quality of the sample are based upon a qualitative visual assessment. However, in this instance, the author has had prior experience performing extractions using the Sealy (1986) method and feels confident in its application to this project.

Another significant concern is that this technique, specifically the use of NaOH, is comparatively harsh. This may result in an artificial reduction in the overall amount of recovered collagen (Chisholm 1989). In contrast, there is little concern that this method may introduce contamination or inadequately remove common archaeological contaminants such as humic acids. Some of the other procedures have been cited for these problems (Schoeninger and Schwarcz 1991). A reduction in collagen yields is problematic in so much that extremely small amounts of collagen may be hard to recover. In addition, if contaminants do remain after extraction and purification, the isotopic composition of collagen in low yield samples may be obscured (Schoeninger et al. 1989). Nevertheless, the Sealy (1986) technique probably provides the best opportunity for removing such contaminants. The potential of reduced collagen yields is thus a fair trade off.

The following section details the procedures employed during the Sealy (1986) collagen extraction and purification methodology. The major stages of this procedure occur in the following order:

1. Section and manually clean the bone samples.
2. Clean specimens in a Hydrosonic bath.
3. De-mineralize samples in 1% HCl.
4. Remove contaminants using 0.125M NaOH solution.
5. Lyophilization (freeze dry samples).

Where necessary, supplementary procedures have also been described.

D.2.1 Technique For Cleaning Bone Samples

1. Manually break the bone up into small chunks of approximately 5-7 g of material using a hammer and/or a small saw.
2. Select a few chunks of appropriate size (or several smaller chunks which yield the same overall mass) to use.
3. Manually and gently clean the samples using distilled water (D-H_2O) and a toothbrush.
4. Clean the samples using a hydrosonic bath for approximately 5-10 minutes (i.e. Branson 1210 Hydrosonic Bath). Repeat if necessary.
5. Allow samples to air dry for approximately 24 hours.

D.2.2 Technique for Preparing 1% HCl Solution

1. Fill an empty container (4 L) with 4 x 990 ml of DD - H_2O.
2. Slowly add 4 x 10 ml of 36% - 38% Concentrated HCl.
   * Remember Acid into Water (A&W rule!) *

D.2.3 Procedure For De-mineralizing Bone Samples Using Hydrochloric Acid (HCl)

1. Select 2-3 g bone chunks from each of the previously cleaned samples (or enough smaller chunks to obtain the appropriate mass).
2. Weigh the selection(s) for each sample using a sample tray on a sensitive scale (milligram range, i.e. Mettler PM460 Delta Range Scale)
   * Zero the sample tray, add the sample, record the weight *
3. Place each sample in a labeled 300 ml beaker
4. Add 100 ml of 1.0 % HCl solution to each beaker
5. Cover each beaker with a watch glass (petri dish)
6. Place samples under a fume hood and allow to react for 48 hour
   * If possible, check each sample at 24 hours, if 25 ml or more solution has reacted away, top up beaker back to 100 ml with 1.0% HCl solution. *
7. After every 48 hour period, decant all solution from each beaker through a strainer leaving the sample within the beaker (Do not attempt to save small flakes of the

sample unless the total sample mass is very small). Replace the old solution in each beaker with new 1.0 % HCl solution (100 ml).

8. Repeat steps 6-7 continuously until collagen extraction is complete.
9. Once collagen extraction is complete, rinse each sample to neutrality using double distilled water (DD-H_2O) and then either store each sample in 100 ml of DD- H_2O or begin NaOH procedure.

* How to determine when collagen extraction is complete *

Qualitative Tests

1. Test the elasticity of the bone structure by gently compressing it within the sample beaker using a sterile probe.

* A spongy structure indicates a lack of mineral (inorganic) content in the bone structure which is equated with collagen extraction *

2. Test the translucency of the bone by holding the sample beaker up to the light. Opaqueness is indicative of remnant mineral content, translucency is characteristic of extracted collagen.

* Note * The elasticity/translucency of any given bone sample once collagen extraction is complete is variable and dependent upon the nature of each specific sample.

Quantitative Tests

1. The HCl used to demineralize bone primarily reacts with the inorganic portion of each sample. Reaction is indicated by the presence of bubbles on the surface of the sample and in solution. If no bubbles occur at any point after the solution is changed and replenished with new HCl, then the reaction is complete as theoretically no mineral content remains for the acid to react with.

   * Note * While the HCl primarily reacts with the mineral content of the bone, continued exposure of the sample to acid once the mineral content has been removed may lead to degradation of the collagen contained within the sample.

2. Changes in sample weight can indicate that reaction is occurring. Determine the weight of the sample at each changing of solution (i.e. weight of the beaker as previously determined, subtracted from the weight of decanted sample plus beaker). At the point that the weight stops changing (decreasing) the reaction is complete.

* Note * Once the mineral content has been removed from the bone, its weight may only briefly stabilize and if continued treatment occurs, the weight may again begin to change as the collagen begins to degrade.

D.2.4 Technique for Rinsing Collagen Samples to Neutrality Using DD - H_2O

1. Decant HCl solution from sample(s) beaker(s).
2. Add approximately 50 ml (or enough to submerge the sample) of DD - H_2O to the beaker.
3. Gently swirl the solution so that the sample becomes saturated and rinsed.
4. Decant the solution through a glass filter system
5. Repeat steps 2 - 3 once then move to step 6.
6. Test the pH of the solution in the beaker using litmus paper, if pH is roughly 6-7 on the pH scale then the sample is neutral, otherwise repeat steps 4-5 continuously until the pH value reaches 6 or 7.
7. Once the sample is neutralized, remove the filter paper from the filter system using sterile tweezers.
8. Rinse the filter into the sample beaker using DD - H_2O (In this way, as little sample material as possible is lost).
9. Sample is now neutralized and ready for next treatment.

Glass Filter System:

a) Filter device is fitted to the top of an Erlenmeyer flask with an outlet.
b) A vacuum hose is fitted to the outlet on the flask and a very light suction is applied.
c) Filter paper is added to the filter device and saturated with DD - H_2O to create a seal between the filter paper and the device.
d) Glass filter system is now ready to use.

D.2.5 Technique for Preparing 0.125M NaOH Solution

1. Measure out 20 g of NaOH pellets using a scale.
2. Add the 20 g of NaOH pellets to an empty 4-5 L container
   * Make sure that magnetic mixing rod is inside the container *
3. Place the container on the Fisher Thermic Stirring Hot Plate (Model 210T).
4. Set the "temperature" dial to off and set the "stir" dial to max.
   * Note * The mixing plate initiates a magnetic field that agitates the magnetic rod inside the container causing it to mix the container contents.
5. Allow mixing to occur for approximately 5 - 10 min.
6. Turn off the plate and store the solution.

D.2.6 Procedure for the Removal of Humic Acid Contaminants From Extracted Collagen Using 0.125% NaOH Solution

1. Decant neutralized samples of all DD- H_2O.
2. Add approximately 100 ml of 0.125% NaOH solution to each sample beaker.
3. Allow each sample to soak for approximately 20 hours.
4. Decant all 0.125% NaOH solution from each sample Beaker.
5. Rinse each sample to neutrality using DD- H_2O.

D.2.7 Procedure for the Transferal of Extracted and Neutralized Collagen Samples From Sample Beakers to Disposable Scintillation Vials

1. Weigh empty labeled scintillation vials without caps and record.
2. Decant neutralized collagen sample beakers of all DD-H_2O.
3. Place an empty disposable 20 ml scintillation vial on a large petri dish (to catch runoff from sample).
4. Use a sterile probe to transfer sample material from beaker into the scintillation vial.
5. Rinse the left over material in beaker into the scintillation vial using DD-H_2O.
   * Do not, however, fill the scintillation vial more than about 1/3 - 1/2 as the liquid must eventually be frozen. *
6. Top up DD-H_2O in each scintillation vial to about 1/3 - 1/2 and cap and label each vial.
7. Place all vials in a freezer and allow to freeze.
   * Once frozen, vials may be stored in freezer indefinitely until freeze drying takes place. *

D.3 Preparation of Organic Samples For Mass Spectrometry

Once collagen has been extracted from the samples, purified and then freeze-dried, it is ready to be combusted. Combustion converts the collagen specimens into gases which must then be purified and separated. These gases are then introduced into a mass spectrometer which measures their isotopic composition and reports isotopic ratios (Boutton 1991). Traditionally, these procedures were carried out manually in a stepwise fashion. More recently, technological advances have allowed for the automation of combustion, gas collection, gas separation and mass spectrometry. Carbon and nitrogen stable isotope ratios are now routinely determined using such automated means (online). Determining stable hydrogen isotope ratios, however, requires a few additional steps that have kept the process largely manual (offline).

The bone collagen samples examined in this study were analyzed to determine their carbon, nitrogen and hydrogen stable isotope compositions. Carbon and nitrogen stable isotope ratios were determined using a largely automated process that will be described in more detail later. The determination of hydrogen isotope ratios was conducted offline. Combustion of each sample was performed in sealed and evacuated vycor tubes that were loaded into a muffle furnace. The resultant gases were then cryogenically separated after loading the sample tubes into a tube sealing vacuum manifold. CO_2 and H_2O were then trapped in separate collection tubes and sealed off. The Hydrogen collection tubes contained zinc alloy. When heated, the H_2O within the tube reacted with the zinc and was reduced to H_2 gas which could then be introduced into a mass spectrometer (see Boutton 1991a, for review).

Although carbon and nitrogen isotope ratios may be measured from CO_2 and N_2 gases that are combusted, distilled and collected in this fashion; it is typically easier to measure these elements using automated systems. The reason why hydrogen isotope ratios have not been typically measured in this fashion is that an additional procedure is required to prepare the samples. The biogeochemistry of hydrogen within substances is unique in that the non-carbon bound hydrogen is labile and tends to replace with environmental hydrogen. Thus, a certain percentage of the hydrogen within bone collagen is not metabolically derived. In order to account for the "interference" caused by environmental hydrogen, an "equilibration" must be performed on each sample

before combustion (see Wassenaar and Hobson 2000, for review). Briefly, once the samples are weighed into vycor tubes, the tubes are attached to a vacuum manifold and evacuated. High temperature steam of known isotopic composition is then introduced into each tube. Exchangeable hydrogen, within the sample, reacts and is replaced with hydrogen from the steam. After a while, the sample tubes are again evacuated and eventually sealed. At this point, the samples may be combusted and resulting gases may be cryogenically distilled. Equilibration allows for calculations to be made that factor out the exchangeable hydrogen within a sample so that the isotopic composition of the non-exchangeable hydrogen may be determined.

The following section describes the procedures used in this project to prepare samples of bone collagen for the determination of hydrogen isotope ratios by mass spectrometry. The major stages involved in this preparation include the following:

1. Weigh samples into vycor combustion tubes
2. Equilibrate samples
3. Combust samples
4. Cryogenically separate gases

Supplementary procedures are also presented where necessary.

D.3.1 Stages of Sample Preparation

1. Place sample into scintillation vial.
2. Remove lipids from sample using a 2:1 Chloroform/Methanol solution (If necessary). Allow to air dry.
3. Weigh out samples into pre-baked (850 degrees) vycor combustion tubes (9mm). Each tube should contain CuO (1.0g) and Cu (0.3g) reagents and each tube should be labeled with the appropriate lab ID (Use a black sharpie to label tube once in the center). Use sample boats (pre-baked 6mm pyrex) or tin cups to contain. sample during transferal into combustion tube (choice is dependant on the nature of samples. i.e. pyrex boats for feathers, tin cups for powdered samples). 7.5mg of sample are ideal. Record sample masses in logbook.
4. Label pre-baked (500 degrees) pyrex gas collection tubes with lab ID. Label each tube twice with a red sharpie. Numbers should be roughly in the center of the tube, but offset from each other. For each sample, a carbon collection tube and a hydrogen collection tube should be labeled. Hydrogen collection tubes require 130mg of standardized zinc as a reagent.
   * If the sample mass is less than the desired amount (i.e. 7.5mg) then the amount

of zinc used in the hydrogen collection tube should be reduced proportionately (i.e. 3.25mg of sample requires 65mg of zinc.)

5. Vycor combustion tubes must be "Necked-down".
6. "Equilibrate" sample combustion tubes.
7. "Seal off" combustion tubes. Record 200ml of equilibration in lab log book.
8. Combust tubes in muffle furnace at 850 degrees for two hours overnight.
9. The next day (ASAP after combustion) remove the combustion tubes from muffle furnace and allow to cool. (Samples should be put to the gas separation line ASAP after combustion, but no longer than 5 days after).
10. Separate gases from combustion tubes on the gas separation line. CO_2 should be collected in empty collection tube, H_2 should be collected in tube containing zinc reagents.
11. "Seal off" tubes and allow to cool.
12. After analysis with a mass spectrometer, enter the results of the mass spectrometry into laboratory log book. Include δD and $\delta^{13}C$ values where applicable, H_2O transducer reading and any relevant notes regarding problems with the sample (i.e. suspect transducer reading, ion gauge not balanced, low significance).

D.3.2 Procedure For Rinsing Lipids From Samples

1. Prepare 300ml of 2:1 Chloroform - Methanol solution (200ml chloroform - 100ml 95% methanol) (Rinses approximately 20 scintillation vials).
2. Use an eyedropper to pipette 2 full eyedroppers worth of solution into each vial.
3. Cap each vial and shake vigorously.
4. Decant each vial.
5. Repeat steps 2-4, two more times (3 total).
6. Allow each vial to air dry, usually over night (keep caps in order for each vial).
   * Solution is carcinogenic, use rubber gloves and fume hood.*

D.3.3 Glass Preparation

VYCOR -Combustion tubes are made from 9mm vycor tubing cut to 18" and then torched with an oxygen flame in the center to seal the ends of two separate tubes at 9".
-Tubes must be baked in the muffle furnace for 2 hours at 850°C before using.
-Vycor tubes must be placed in an iron casting for baking.

6mm Vycor Sample boats: Cut from 6mm vycor tubing at approximately 5cm lengths by scoring and breaking the tubing. Boats are pre-baked in a pyrex beaker in the muffle furnace at 500°C for 2 hours.

PYREX -Collection tubes are made from 6mm pyrex tubing cut to 12" and then torched with an oxygen flame in the center to seal the ends of two separate

tubes at 6".
-Tubes must be baked in the muffle furnace for 2 hours at 500°C before using.
-Pyrex tubes must be placed in a pyrex beaker for baking.

D.3.4 Procedure For Weighing Out Samples Into Combustion Tubes

Solid Samples:

* Must maintain a sterile environment throughout procedure.

1. Place a weighing paper on the scale.
2. Place a 6mm vycor sample boat on the scale.
3. Close scale doors.
4. Zero the scale.
5. Using tweezers, forceps and scissors; place sample in the sample boat (do not directly handle either the sample or the sample boat).
6. Weigh sample boat and sample with scale doors closed. Allow scale to equilibrate, sample mass should be approximately 7.5mg (if sample is < 7.5mg use all available mass; if sample amount is > 7.5mg reduce the amount until a mass of 7.5mg is achieved. *Save excess sample in initial scintillation vial.
7. Record sample mass.
8. Place sample boat into corresponding vycor combustion tube (labeled with lab ID and containing 1.0g of sieved CuO and 0.3g of Cu reagents). If necessary push sample boat to the base of the tube using a sterile probe.
9. Wipe all utensils with a kimwipe. Replace weighing paper if necessary and clean off the scale baseplate with a brush prior to weighing the next samples.

Powdered Samples:

* Must maintain a sterile environment throughout procedure.

1. Select an appropriate size of tin cups to use for procedure (usually 4x6mm).
2. Using forceps, place tin boat on the scale.
3. Close scale doors.
4. Zero the scale.
5. Using forceps, remove tin cup from the scale and place it inside the appropriate sized aperture in the tin cup weighing baseplate (metal baseplate stored in a case near the scale).
6. Using a sterile probe, transfer some of the powdered sample into the tin cup (some will spill onto the baseplate).
7. Using forceps, gently remove the tin cup from the baseplate aperture and gently shake the powder down into the base of the cup by gently picking up the cup and dropping it several times against the baseplate with the tips of the forceps remaining within the rim of the cup to prevent it from spilling.

8. Place the tin boat with sample on the scale. The desired sample mass is 7.5mg. If more sample is needed, use the above procedures to add more. If sample needs to be removed from the cup, use forceps to gently pour some of the sample out until the desired mass is reached.
9. Record sample mass.
10. Use forceps to gently pinch off the top of the tin boat (This helps to prevent static spray of the sample once inside a combustion tube).
11. Place tin cup into corresponding vycor combustion tube (labeled with lab ID and containing 1.0g of sieved CuO and 0.3g of Cu reagents). If necessary push the tin cup to the base of the tube using a sterile probe (be gentle as tin cups are very fragile).
12. Wipe all utensils with a kimwipe. Clean off the scale baseplate with a brush prior to weighing the next samples. Dump out excess sample from the tin cup weighing baseplate as well.
13. When all samples have been weighed, clean all utensils and the tin cup weighing baseplate with ethanol. Allow to air dry.

* Weighing out standards is the same procedure for both solid and powdered samples. The standard used at the NHRC for H samples is Polyethylene Foil (PEF 1). For standards, the required mass is 2.5g. The standard is placed and weighed in a 6mm vycor sample boat using the same procedures described for weighing out solid samples. The standard plastic is cut from a larger sheet and manipulated with scissors and forceps (Do not touch the plastic!).

* Once the samples and standards have been weighed out, the combustion tubes must be "necked down" using an oxygen flame. "Necking down" involves softening an area of the tube about 2.5 inches down from its opening and stretching the tube to create a 1 - 2" long neck (thinned to about 1-2mm in diameter). "Necking down" prevents samples from being sucked out of the combustion tube during equilibration and allows for the tubes to be sealed off with an oxygen flame after equilibration.

D.3.5 Equilibration

1. Prepare equilibration line
   a. Fill the two online LN_2 (Liquid Nitrogen) traps with LN_2.
   b. Load "necked-down" combustion sample tubes onto the manifold.
   c. Turn on heat. (both temperature gauges).
      Upper gauge (temp) 95°C ± 1
      Lower gauge (temp) 135°C ± 1
   d. Make sure that diffusion and backing pumps are on (red lights on the blue box indicate that they are on).
   e. Open the 2 main vacuum pump valves.
      f. Slowly open the on-line manifold septum valves in order from 1-10 (Open valves 1.5 full turns).

g. Slowly open the three vacuum stop cocks (valves) in the following order: 2, 3, 1.
h. Open the "roughing pump". Allow the system to pump down to 10^{-1} torr, then switch to the "backing pump".
i. Open the diffusion pump (Never allow both the roughing pump and the diffusion pump to be open at the same time).

2. Allow the system to pump down to 0.0×10^{-4} torr, or close to this for 1 hour.
3. After 1 hour, check the pressure.
4. Slowly close all manifold septum valves in order from 1-10.
5. Slowly close the three vacuum stop cocks (valves) in the following order: 2, 3, 1.
6. Get new equilibration water (-135‰).
7. Clear out syringe (three times).
8. Quickly inject 200ml of equilibrated water into each septum (1-9), (septum 10 is usually a standard and thus, is not injected - any standard tubes or empty, unused septums are not injected, only samples). The needle hole should face down during injection.
9. Allow the system to equilibrate for two hours.
10. After two hours, slowly open all manifold septum valves in order from 1-10 (Slowly open the first valve and watch for steam release).
11. Slowly open the three vacuum stop cocks (valves) in the following order: 2, 3, 1 (When opening valve 2, open slowly and wait for a steam reaction with the connecting LN_2 trap which will result in sizzling and bubbling, then open valves 3, 1.
12. Allow the system to pump down for 1 hour.
13. Slowly close off manifold septum valves in order from 1-10.
14. Slowly close off the three vacuum stop cock valves in the following order: 2, 3, 1.
15. Turn off heat (both upper and lower gauges).
16. Close the diffusion pump, and make sure that the backing pump is on.
17. Close the two main pump valves.
18. Allow system to cool.
19. "Seal off" and remove sample combustion tubes.

Note: Throughout equilibration, periodically check the LN_2 level in both large traps and top up if necessary. Also check the pump pressure and temperature gauges.

D.3.6 Gas Separation Line Procedures

1. Prepare Gas Separation Line
   a. Fill on-line large dewar housing water trap with LN_2.
   b. Turn on heating tape
   c. Turn on both pressure gauges.
   d. Prepare dry ice slush
2. Evacuate both loops (to about 10^{-2} torr).

- During evacuation: Load the sample vessels; attaching breakseal vessel containing the combusted sample on the left and carbon tube on the right.

3. Close valves to the loops and slowly open valves to the sample tubes for evacuation.
4. Place the short dewars around the loops and fill them with LN_2.
5. Once the sample vessels have pumped down, close valves 1 and 2.
6. Crack the sample inside the breakseal vessel, then open valves 1 and 3 to let gas transfer into 1st loop, look for the transfer before continuing.
7. Heat the breakseal vessel for 1 minute - Low heat.
8. Open horizontal valve between the loops and record the transducer reading… this represents the N_2 gas which must be evacuated… close valve to CO_2 tube and then open the 5th valve to evacuate N_2.
   - While waiting for N_2 to pump out, fill up dewar with hot water
9. Close all valves.
10. Remove dewar from 1st loop and place it around the CO_2 tube.
11. Place dewar of dry ice slush around the 1st loop.
12. Replace breakseal vessel with collection tube containing zinc (hydrogen tube).
13. Open valve between the loops to allow CO_2 to separate from H_2O.
14. Open Valves 1 and 2 to pump out zinc tube.
15. Heat zinc tube with high heat using the heating gun for 1 minute (minimum), or until the tube is completely pumped down (evacuated to 10^{-2} torr).
16. Close valves 1, 2 and 3.
17. Replace the dry ice slush dewar with hot water dewar.
18. Transfer the dewar from the 2nd loop to the zinc collection tube.
19. Open valves 1 and 3, to allow H_2O to transfer into zinc tube.
20. Heat the 2nd loop with high heat using the heating gun until the transducer reading stabilizes.
21. Record the transducer reading indicating the CO_2 pressure.
22. Open and close the 5th valve to fill the glass space between valves 5, 6 and 7 with CO_2, then open and close the valve (valve 7) to the CO_2 tube to allow the gas to transfer ---- repeat this step once.
23. Wait 7 minutes before sealing the tubes (To allow for H_2O transfer).
24. Top off dewars around the two collection tubes with LN_2.
25. Raise dewars up the sample tubes before sealing off.
26. Use oxygen torch to carefully seal off the sample collection tubes
27. Collect, bind and label CO_2 series collection tubes. Collect bind and label H_2 series collection tubes (usually 9 sample tubes + 1 standard tube per series). Each series of tubes should be bound with a rubber band and labeled with a sticky note held fast to the tubes with the rubber band.

* The following format should be used as an example of how to label the tubes:

11000 - 11009
S11015
WASSENAAR

First the sample series as denoted by the Lab ID's, second the Lab ID for the standard utilized in the series, third the client ID.

28. Store the CO_2 series tubes in the sample cabinet. H_2 series tubes must be stored in the freezer.
29. Close Down Gas Separation Line
    a. Cap dry ice slush dewar and store in freezer.
    b. Consolidate all dewars containing LN_2 into one large dewar, cap and leave on counter.
    c. Evacuate both loops on the line.
    d. Turn off pressure and transducer gauges.
    e. Turn off heating tape.
    f. Turn off gas and oxygen spouts

D.3.7 Preparation of Dry Ice Slush

1. Get dewar containing old slush mixture from freezer.
2. Use the CO_2 siphon tank to get a full block of dry ice. (If a lot of dry ice is still present in the mixture, use less than a full block).
3. Dump the dry ice block into the dry ice rubber receptacle and use the plastic scoop to break it up.
4. * If required, add more ethanol to the dewar (Top it up to the small circle on the interior of the dewar).
5. Add dry ice slowly to the dewar (until the temperature is significantly decreased, the dry ice will quickly dissipate and react with the ethanol). Add dry ice until the solution level is near the top of the dewar and the consistency is "like a dry slush".
6. Cap dewar and set aside until needed.

   * Temperature of slush should be near - 78°C, use thermocouple to gauge.

D.4 Mass Spectrometry

D.4.1 Introduction

The basic function of a mass spectrometer is to separate isotopes based on their masses. This allows for the measurement of relative abundances of various isotopes within a substance. Isotope geochemists use these abundances as tracers of geologic and biological processes, geochronometers (dating techniques) and paleothermometers (formation and substance temperatures). There are several types of mass spectrometer. A technological distinction is made between mass spectrometers that are used in stable isotope geochemistry and those used for radiogenic isotope geochemistry. The design of mass spectrometers used to study the stable isotope geochemistry of light elements such as hydrogen, oxygen, carbon, nitrogen and sulphur is based on the Nier mass spectrometer developed following the Second World War (Nier 1947). This design has been evolving over the last 40 years and is still in the process of refinement.

Lower mass isotopes are usually measured using an isotope ratio mass spectrometer (IRMS). The various compounds to be analyzed are usually introduced into the instrument as purified gas samples (i.e. H_2, CO_2, N_2). The gas isotope ratio mass spectrometer (GIRMS) ultimately measures the mass to charge (m/z) ratio of isotopic species within a substance, in the form of ions. Because the ratios are determined internally, the researcher never knows the absolute value of anything. Gas injected into the mass spectrometer is ionized and a magnet is used to project and split the ionized isotopes into two or more beams with distinct trajectories. The heavier isotopes form large ion beams and the lighter isotopes form smaller ones (see Boutton 1991a).

Although it is the mass differences between the isotopes that allow for them to be separated, it is the relative differences in voltage between the ion streams that are ultimately measured to produce a ratio. The absolute voltage measurements for two distinct substances may be vastly different, but if the voltage difference between the generated ion streams are the same in each case, the mass spectrometer will report the same isotope ratio. For example; the absolute voltages from different ion streams resulting from sample A could be reported as 8 and 4, while sample B is 6 and 2. Although the absolute voltages differ in each case, the differential voltage in both

instances is 4. Thus the mass spectrometer would report the same outcome in both cases. All measurements are relative and not absolute. This distinction is important as the absolute measurements associated with each different stream can vary substantially amongst different samples due to a variety of factors including differences in the initial mass of each specimen.

IRMS are composed of four major components: an inlet system, an ion source, a mass analyzer, and a series of ion detectors (Katzenberg 2000). Compounds are introduced into the instrument as pure gas samples, through an inlet system at the front end of a flight tube which directs the gas into the ion source. Until recently, most stable isotope research involving collagen required combustion in sealed tubes (as described previously). After combustion the resultant gases were then separated offline and later fed individually into the mass spectrometer through a manual inlet system. An advance in manual inlet technology was the development of the dual-inlet system in which the sample gas and a reference gas are introduced into the IRMS ion source through matched, but separate capillaries. This innovation helped to reduce fractionation of the gases.

In a dual inlet system, variable volume bellows allow for control of the size of the ion beams and help to equalize the gas pressure avoiding excess fractionation. Change over valves allow for entrance of either the sample or reference gas into the ion source. Similarly, either may be vented at any time which may help to equalize pressure in the system. Once in the mass spectrometer, the input gas enters the ion source and some of the gas molecules are ionized by electron bombardment. This allows for them to be controlled and focused into a beam. Once ionized, the gas molecules possess a charge (usually positive for GIRMS). A negative charge generated within the mass spectrometer creates an accelerating potential that propels the positive ions down a flight tube and into the mass analyzer.

The mass analyzer separates the ion beam into several smaller beams by passing it at a constant speed between the poles of a magnet held at a constant strength. The separation of one ion beam into several smaller beams according to mass yields a "mass spectrum". This concept is analogous to the use of a prism to separate white light into its constituent spectra via wavelength. The trajectory of the ions passing through the

magnetic field result as a function of the mass and energy of the ions themselves. Ions of lower mass have less momentum than do those of higher mass and are thus deflected to a greater degree. Each ion beam then strikes a different Faraday cup collector where the individual beam intensities are measured and assessed (see Boutton 1991a and Katzenberg 2000).

Modern instruments now interface combustion furnaces and gas elemental analyzers (EA) with mass spectrometers to simplify and ease the conversion of the sample into a gaseous form. In such a setup, collagen is weighed into inert tin or silver sample cups, which are then placed into an automated sample tray. The revolving tray drops samples into the furnace where N_2, CO_2 and H_2O are produced. The combustion products then flow through a reduction tube where the substance is reduced to a purified elemental state. This purification is achieved through gas chromatography and a small quantity is then transported to the mass spectrometer by capillary tubing where the isotopic composition can then be determined. A commonly used example of this technology is the automated nitrogen/carbon analyzer (ANCA) mass spectrometer.

Interfaces such as these are commonly employed with continuous flow isotope ratio mass spectrometers (CF-IRMS). In continuous flow systems, instrumental fractionation is less and thus samples may be run with only sporadic testing of the standard gas (approximately every 10 samples). The system is more linear than dual inlet. Once the sample gas has been produced, pulses of it are injected into a "carrier gas" through which they are then transmitted into the mass spectrometer. The carrier gas is used to pressurize the system and create a "viscous flow" into which the sample gas can be introduced. For the most part, the carrier gas is inert (i.e. a noble gas such as He) and thus, does not become ionized within the system. It also has a different mass from the sample gas and can therefore be removed. Because of design and automation, continuous flow systems tend to be faster, allowing for more samples to be run. Additionally, this technique usually requires significantly less sample mass than do others.

Stable isotope ratios for all of the samples analyzed in this project were determined at the Stable Isotope Laboratory at the National Hydrology Research Centre in Saskatoon. D/H ratios were measured on a Micromass Optima dual inlet isotope ratio

mass spectrometer. The same instrument was then reconfigured for continuous flow and coupled with an elemental analyzer for the analysis of $^{13}C/^{12}C$ and $^{15}N/^{14}N$ ratios. As previously noted, determination of the isotopic composition of samples by EA analysis, does not require laborious offline preparation.

Typically, samples for EA-analysis require less mass than do those prepared offline. The mass required to run organic samples is usually about 1mg, although this will depend on the expected concentrations of carbon and/or nitrogen within the sample. Each specimen (usually powdered) is weighed into a tin capsule (3.5 x 5 mm), folded at least twice and placed into the automated sample carousel. Prior to combustion, all air must be removed from the capsule (i.e. crush it!). The carousel compartment designation, sample mass, sample or lab ID and any other relevant information are recorded. Depending on sample composition, any number of different standard materials will be tested along with the samples. Usually, one standard is run for about every 10 samples. For this project, Pugel (Princeton University Gel) was used as the carbon/nitrogen standard. The mass of each Pugel standard was approximately 1mg.

D.4.2 Correction Calculations

When samples are run through a mass spectrometer to obtain isotopic ratios, standards of known isotopic value are tested along side them. These internal lab standards should be differentiated from the international ones used to standardize results for purposes of comparison. The values initially reported following mass spectrometry of a given sample are the measured isotopic composition of that sample relative to an international standard of known value. For reporting purposes, all isotopic ratios for a given element are standardized through comparison to the relevant international standard for that element. On each scale, the international standard, regardless of actual isotopic composition, is defined as 0‰ and all other isotopic ratios for that element are reported in reference to that standard value. Isotopic ratios of carbon, nitrogen and hydrogen were measured for all samples in this study and the corresponding international standards for each are as follows:

Carbon: PeeDee Belemnite (PDB)

Nitrogen: Atmospheric Nitrogen Gas (AIR)
Hydrogen: Vienna Standard Mean Ocean Water (VSMOW)

In contrast, internal lab standards are used to correct for "isotopic drift" that may result during the mass spectrometry of samples.

The process of measuring the isotopic composition of a substance via mass spectrometry induces fractionation which slightly alters the isotopic ratio within the analyzed specimen. The degree of this fractionation may be variable and can be affected by several factors, including the mass of the analyzed sample, pressure within the system itself, the configuration of the system and the degree of ionization that occurs. To correct for this, lab standards of known value are periodically tested during a run of experimental samples. By gauging the amount of variation which occurs between the values obtained for these standards and the predetermined actual value of these standards, corrections for the experimental samples being tested may also be calculated. Usually, one standard is run for about every ten samples that are tested during an experimental set.

The laboratory standards utilized during this project were Pugel (Princeton University Gel) for carbon and nitrogen and PEF-1 (Polyethylene Foil) for hydrogen. The isotopic composition of these standards are as follows:

Pugel: $\delta^{13}C = -12.6$ ‰
$\delta^{15}N = 5.6$ ‰
PEF-1: $\delta D = -100.3$‰

Any deviation from these ideals, occurring amongst samples of each standard throughout an experimental run, can be used to calculate a correction factor which may be used to adjust the measured values of an experimental set. An example of this is provided below.

Table D1 shows the measured isotopic composition of a series of standards run during the analysis of carbon and nitrogen isotope compositions for a limited set of samples analyzed during this study. The mean measured $\delta^{13}C$ and $\delta^{15}N$ values for each of these standard samples were calculated. The average difference between these means

and the expected $\delta^{13}C$ and $\delta^{15}N$ values for Pugel were used to correct all measured values for each experimental specimen.

Table D1. Variation Among the Measured Isotopic Composition of Pugel Standards During Analysis of a Select Set of Experimental Samples.

Standard	$\delta^{13}C$ (‰)	$\delta^{15}N$ (‰)
Pugel	-12.38	5.75
Pugel	-12.5	5.89
Pugel	-12.53	5.87
Pugel	-12.64	5.8
Pugel	-12.47	5.9
Pugel	-12.53	6
Average	-12.5083	5.868333
Stdev	0.085186	0.086583
Average Difference	-0.11167	-0.26833

The average measured $\delta^{15}N$ of the tested Pugel standards was 5.87‰. The average variation between this measured mean and the actual nitrogen isotope composition of Pugel (5.6‰) was determined to be -0.27‰. This value is the correction factor used to adjust all of the measured results from this sample run. It is added to all of the experimentally determined values. As an example, the measured nitrogen isotope ratio for sample j5 is 7.04‰. The corrected value is 6.8‰ (7.04 + -0.27). The conversions for the rest of the samples in this run are presented in Table D2.

Table D2. Correction of the Measured Isotopic Composition of Collagen Samples During a Selected Run.

Sample	Measured Delta		Corrected Delta	
	$\delta^{13}C$	$\delta^{15}N$	$\delta^{13}C$	$\delta^{15}N$
j4	-22.86	5.82	-23.0	5.6
j5	-22.81	7.04	-22.9	6.8
j6	-22.09	4.36	-22.2	4.1
j7	-28.96	2.85	-29.1	2.6
j8	-26.86	8.06	-27.0	7.8
j9	-28.51	8.01	-28.6	7.7
j10	-27.07	2.4	-27.2	2.1

The corrected δ values for carbon and nitrogen are reported to one significant digit in deference to the precision limitations of the equipment used to measure them. The measured $\delta^{13}C$, $\delta^{15}N$ and δD results for each sample analyzed during this project were corrected for isotopic drift and are reported in Table D3. Correction of hydrogen isotope values involve additional steps including an adjustment based upon the type of zinc used in the distillation of pure hydrogen gas from the extracted sample water, and corrections for hydrogen exchange which are detailed in the next section.

Table D3. Corrected Carbon and Nitrogen Stable Isotope Ratios For All Specimens

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	$\delta^{13}C$ (‰)	$\delta^{15}N$ (‰)	$\delta^{2}D$ (‰)	Comments
A1	12450	Heron Eden Site (EeOi-11), Cody Complex, 9150 BP	-19.3	6.3	\	
A2	12451		-19.8	6.1	-132	
A3	12452		-19.1	7.9	-149	
A4	12453		-17.3	5.5	-145	
A5	12454		-19.2	5.9	-133	
A6	12455		-19.8	5.5	-132	
A7	12456		-16.9	6.2	-141	
A8	12457		-19.5	6.3	-136	
A9	12458		-19.2	6.5	-127	
A10	12459		-20.0	6.2	-137	
A11	12460		-20.1	5.8	-134	
A12	12461		-17.4	6.3	-143	
C1	12468	Norby Site (FaNq - 56), Mummy Cave, 7050 BP	-15.7	6.8	-138	
C2	12469		-17.9	6.2	-150	
C3	12470		-17.2	6.5	-138	
C4	12471		-19.5	7.2	-124	
C5	12472		-18.4	8.2	-130	
C6	12473		-16.5	7.3	-135	
C7	12474		-19.2	7.7	-129	
C8	12475		-16.4	7.3	\	
C9	12476		-17.6	7.1	-139	
C10	12477		-17.7	8.9	-158	
C11	12478		-18.9	6.9	-129	
C12	12479		-19.4	8.4	-123	
C13	12480		-19.3	8.2	-123	
C14	12481		-18.4	8.7	-112	
B1	12462	Gowen Sites (FaNq - 32), MummyCave, 5850 BP	-19.2	7.1	-145	
B2	12463		-19.3	8.9	-132	
B3	12464		-18.4	8.3	-134	
B4	12465		-18.8	7.8	-142	
B5	12466		-19.5	9.8	-130	
B6	12467		-20.0	7.3	-134	
D1	12482	Amisk (FbNp - 17), Oxbow, 4350 BP	-19.9	8.2	-131	
D2	12483		-19.2	7.6	-127	
E1	12484	xbow, 4200	-19.1	6.4	-124	
E2	12485		-21.6	7.4	-140	
E3	12486		-20.5	8.8	-142	
E4	12487		-19.1	7.7	-146	
E5	12488		-19.2	8.2	-143	

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	$\delta^{13}C$ (‰)	$\delta^{15}N$ (‰)	$\delta^{2}D$ (‰)	Comments
E6	12489	Harder Site (FbNs - 1), O BP	-16.5	6.4	-125	
E7	12490		-19.0	7.8	-131	
E8	12491		-19.2	7.5	-141	
E9	12492		-19.3	6.8	-126	
E10	12493		-19.7	8.7		
E11	12494		-19.6	7.4	-140	
E12	12495		-18.8	7.3	-143	
E13	12496		-21.9	8.6	-150	
E14	12497		-19.9	8.6	-162	
E15	12498		-19.7	8.3	-149	
F1	12499	Thundercloud Site (FbNp - 25), McKean, 3400 BP	-19.5	8.1	-148	
F2	12500		-18.5	7.2	-132	
F3	12501		-20.2	7.2	-134	
G1	12502	Fitzgerald Site (ElNp - 8), Besant, 1550 BP	-17.5	7.8	-116	
G2	12503		-18.5	7.4	-130	
G3	12504		-18.6	8.1	-116	
G4	12505		-16.1	7.9	-239	
G5	12506		-19.6	8.2	-95	
G6	12507		-16.4	6.9	-151	
G7	12508		-19.1	8.5	-130	
G8	12509		-18.3	7.7	-130	
G9	12510		-18.8	8.7	-213	
G10	12511		-18.0	7.8	-119	
G11	12512		-16.1	7.1	-111	
G12	12513		-18.5	8.4	-122	
G13	12514		-17.0	7.4	-117	
G14	12515		-16.2	7.6	-119	
G15	12516		-16.2	6.5	-131	
H1	12517	Tschetter Site (FbNr - 1), Prairie Side Notched, 1050 BP	-19.8	7.0	-141	
H2	12518		-19.7	5.2	-132	
H3	12519		-19.6	6.3	-126	
H4	12520		-17.7	6.5	-120	
H5	12521		-17.6	6.1	-119	
H6	12522		-19.4	7.5	-126	
H7	12523		-18.1	6.8	-129	
H8	12524		-19.3	7.0	-120	
H9	12525		-18.9	6.4	-123	
H10	12526		-20.1	5.6	-186	
H11	12527		-18.8	6.3	-133	
I1	ues Are regates	Oklahoma, Modern	-13.3	3.2	-73	
I2			-15.7	4.9	-70	
I3			-15.0	3.1	-87	
J1		Saskatoon, Modern	-21.6	5.2	-135	

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	$\delta^{13}C$ (‰)	$\delta^{15}N$ (‰)	$\delta^{2}D$ (‰)	Comments
J4	*Val Aggr	Saskatoon, Modern	-23.0	5.6	-146	
J5		Yorkton, Modern	-22.9	6.8	-130	
J6			-22.2	4.1	-130	
Dietary Samples						
I4	*Values Are Aggregates	Oklahoma, Modern	-18.2	14.1	-105	Stomach Contents
J2		Saskatoon, Modern 2001	-26.5	2.5	-130	Feed
J3			-26.8	4.3	-172	Grass
J7		Saskatoon, Modern 2002	-29.1	2.6	-182	Grass
J8			-27.0	7.8	-155	Feed
J9		Yorkton, Modern 2002	-28.6	7.7	-198	Stomach Contents
J10			-27.2	2.1	-181	Grass

D.4.3 Hydrogen Exchange Calculations

A final correction must be performed for any hydrogen isotope ratios that are determined. This is necessary because of the complications of hydrogen exchange. Equilibration procedures are designed to control for this exchange and allow for calculations that may determine the δD of the non-exchangeable hydrogen (δD_n) within a sample. Performing such calculations requires that a researcher know the overall percentage of hydrogen within a given substance that is exchangeable. This may be determined experimentally using a static equilibration technique with steam having a wide range of hydrogen isotopic values (see Wassenaar and Hobson 2000, for review).

Briefly, the ability to determine the percentage of exchangeable hydrogen relies upon the fact that this proportion remains constant within a given material. Thus, when samples of a substance are equilibrated with waters of differing isotopic composition, the resulting isotopic composition of each sample will vary in a linear fashion according to the isotopic composition of the equilibration water. A complication is that the magnitude of this relationship is also temperature dependant. This is because hydrogen exchange between substances results in a temperature dependant isotopic fractionation. In the end, the hydrogen isotope composition of any specimen results from both the percentage of hydrogen that is exchangeable within the sample and the degree of temperature dependant isotopic fractionation that occurs during exchange.

Solving for the amount of exchangeable hydrogen within a given substance requires that one know the fractionation factor for the material at a given temperature. Unfortunately, this is not easily determined for complex organic materials (Hobson *et al.* 1999). Thus, an estimated range for the fractionation factor may be employed based upon typical values for complex organic substances (Hobson *et al.* 1999, Wassenaar and Hobson 2000). For this project, a simple two-end member isotopic equilibration procedure was used to calculate the proportion of exchangeable hydrogen (f_e) in bone collagen, using samples from modern Saskatchewan bison. This was accomplished by comparing the isotopic composition of different samples of an individual animal's collagen tissue (δD_T) that were equilibrated with waters of different isotopic composition (δD_W) using the following equation:

$$f_e = \frac{(\delta D_{TA} - \delta D_{TB})}{(\delta D_{WA} - \delta D_{WB})\,(1 + \epsilon_{x\text{-}w}/1000)}$$

The A and B subscripts refer to equilibration waters of widely different δD composition. Each individual specimen was tested with three pairs of static equilibrations to determine the average percentage of exchangeable hydrogen (i.e. -135‰ vs. +115‰, +115‰ vs. +525‰, -135‰ vs. +525‰). A provisional estimate of the equilibrium isotopic fractionation factor ($\epsilon_{x\text{-}w}$) of 80‰ was used, with a sensitivity analysis ranging between +60‰ and +100‰ (Wassenaar and Hobson 2000). This provided a series of estimates for the proportion of exchangeable hydrogen (Table D4.)

Table D4. Calculations of the Proportion of Exchangeable Hydrogen Within Modern Saskatchewan Bison Bone Collagen Using Static Equilibration With Waters of Varying δD.

Modern Bone Samples	Measured Parameters (VSMOW)				δD_n (VSMOW) (‰)			% H exchangeability (of total H)		
	δD_{TA}	δD_{TB}	δD_{WA}	δD_{WB}	ϵ = 60‰	ϵ = 80‰	ϵ = 100‰	f_e @60‰	f_e @80‰	f_e @100‰
Saskatoon	-135	-13	-135	525	-146	-149	-152	0.17	0.17	0.17
(n = 2)	-86	-8	115	525	-145	-148	-151	0.18	0.18	0.17
	-146	-81	135	115	-166	-171	-176	0.25	0.24	0.24
	Mean:				-152 ± 12	-156 ± 13	-160 ± 14	0.20 ± 0.04	0.20 ± 0.04	0.19 ± 0.04
Yorkton	-130	9	-135	525	-142	-146	-149	0.20	0.20	0.19
(n = 2)	-79	17	115	525	-153	-157	-162	0.22	0.22	0.21
	-130	-76	-135	115	-142	-146	-150	0.20	0.20	0.20
	Mean:				-146 ± 6	-150 ± 7	-154± 7	0.21 ± 0.01	0.20 ± 0.01	0.20 ± 0.01
	Regression:									**20.0 ± 2.5**

Definitions

δD_{TA}	measured total A	ϵ = 60	fractionation factor
δD_{TB}	measured total B	ϵ = 80	fractionation factor
δD_{WA}	water A	ϵ = 100	fractionation factor
δD_{WB}	water B	f_e	fraction of exchangeable
δD_n	non-exchangeable H (calculated)	Regression	% exchange using all data

Estimates of the proportion of exchangeable hydrogen resulting from each test were eventually averaged for use in deriving the isotopic composition of non-exchangeable hydrogen (δD_n) in all modern and prehistoric samples of bison bone collagen. The average percentage of exchangeable hydrogen in collagen, as determined from samples of modern Saskatchewan bison bone, is 20.0 ± 2.5. This proportion is consistent with other published estimates for various biological tissues (Schoeller *et al.* 1986, Cormie *et al.* 1994, Hobson *et al.* 1999). The percentage of non-exchangeable hydrogen in a sample may be calculated using the following equation:

$$\delta D_n = \frac{(\delta D_T - (f_e * \alpha * \delta D_W)}{1 - f_e}$$

where $\alpha = 1.080$. This value is a temperature dependant isotopic fractionation factor, based on the recommended equilibrium isotopic fractionation factor of 80‰, by the following relationship:

$$\alpha = 1n^{-1} (\epsilon_{x\text{-}w} /1000)$$

Once the proportion of exchangeable hydrogen has been determined for a substance, δD of non-exchangeable hydrogen in samples of the same material may be calculated using the same pre-determined exchangeable hydrogen percentage. All subsequent specimens may be equilibrated using a single water of known isotopic value. For this project, all subsequent equilibrations utilized water (δD_W) with a hydrogen isotope composition of -135‰.

The results of all of the bone collagen hydrogen exchange correction calculations are presented in Table D5. All modern and prehistoric Saskatchewan area bison samples were corrected using an exchange factor of 20.0 ± 2.5. The results for the modern Oklahoma area bison and all dietary samples collected with this thesis, are also presented here. These corrected values are the final reportable δD values for each specimen in this project. Hydrogen exchange was considered separately for the Oklahoma bison. Static equilibration of the Oklahoma samples indicated a similar but

distinct proportion of exchangeable hydrogen (15.2 ± 4.3) which was used only to calibrate the Oklahoma bone collagen results (Table D6). The reasons for this difference are not immediately clear. Bison dietary samples were also collected for each of the modern Saskatchewan and Oklahoma sample groups. Each dietary specimen underwent its own series of static equilibration tests to determine the relevant proportion of exchangeable hydrogen in each case. These results are presented in Tables D7 - D9.

Finally, all of the modern specimens, both collagen and dietary samples, were tested multiple times during the static equilibration procedure. As a result, Table D10 presents the results of these various trials using waters of different δD composition. The hydrogen isotope composition of each modern sample reported in this thesis is derived from an aggregate of all of the ratios determined during multiple equilibrations with water of -135‰ δD composition. The values in this table are uncorrected for hydrogen exchange and have only been adjusted for isotopic drift and zinc effects.

Table D5. Hydrogen Exchange Corrections For All Samples.

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	δD (‰) Equilibration Waters (200 µl)	% Exchangeable Hydrogen	Fractionation Factor	δD (‰)	Comments
A1	12450	Heron Eden Site (EeOi - 11), Cody Complex, 9150 BP	-135.0	20.0	1.08	36	
A2	12451		-135.0	20.0	1.08	-126	
A3	12452		-135.0	20.0	1.08	-147	
A4	12453		-135.0	20.0	1.08	-142	
A5	12454		-135.0	20.0	1.08	-128	
A6	12455		-135.0	20.0	1.08	-126	
A7	12456		-135.0	20.0	1.08	-137	
A8	12457		-135.0	20.0	1.08	-131	
A9	12458		-135.0	20.0	1.08	-120	
A10	12459		-135.0	20.0	1.08	-132	
A11	12460		-135.0	20.0	1.08	-129	
A12	12461		-135.0	20.0	1.08	-140	
C1	12468	Norby Site (FaNq - 56), Mummy Cave, 7050 BP	-135.0	20.0	1.08	-134	
C2	12469		-135.0	20.0	1.08	-148	
C3	12470		-135.0	20.0	1.08	-134	
C4	12471		-135.0	20.0	1.08	-117	
C5	12472		-135.0	20.0	1.08	-124	
C6	12473		-135.0	20.0	1.08	-130	
C7	12474		-135.0	20.0	1.08	-123	
C8	12475		-135.0	20.0	1.08	36	
C9	12476		-135.0	20.0	1.08	-135	
C10	12477		-135.0	20.0	1.08	-158	
C11	12478		-135.0	20.0	1.08	-123	
C12	12479		-135.0	20.0	1.08	-115	
C13	12480		-135.0	20.0	1.08	-115	
C14	12481		-135.0	20.0	1.08	-102	
B1	12462	Gowen Sites (FaNq - 32), MummyCave, 5850 BP	-135.0	20.0	1.08	-142	
B2	12463		-135.0	20.0	1.08	-126	
B3	12464		-135.0	20.0	1.08	-129	
B4	12465		-135.0	20.0	1.08	-139	
B5	12466		-135.0	20.0	1.08	-124	
B6	12467		-135.0	20.0	1.08	-129	
D1	12482	Amisk (FbNp - 17), Oxbow, 4350 BP	-135.0	20.0	1.08	-125	
D2	12483		-135.0	20.0	1.08	-120	
E1	12484	4200	-135.0	20.0	1.08	-117	
E2	12485		-135.0	20.0	1.08	-136	
E3	12486		-135.0	20.0	1.08	-139	

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	δD (‰) Equilibration Waters (200 µl)	% Exchangeable Hydrogen	Fractionation Factor	δD (‰)	Comments
E4	12487	Harder Site (FbNs - 1), Oxbow, BP	-135.0	20.0	1.08	-144	
E5	12488		-135.0	20.0	1.08	-140	
E6	12489		-135.0	20.0	1.08	-118	
E7	12490		-135.0	20.0	1.08	-125	
E8	12491		-135.0	20.0	1.08	-137	
E9	12492		-135.0	20.0	1.08	-119	
E10	12493		-135.0	20.0	1.08	36	
E11	12494		-135.0	20.0	1.08	-136	
E12	12495		-135.0	20.0	1.08	-140	
E13	12496		-135.0	20.0	1.08	-148	
E14	12497		-135.0	20.0	1.08	-163	
E15	12498		-135.0	20.0	1.08	-147	
F1	12499	Thundercloud Site (FbNp - 25), McKean, 3400 BP	-135.0	20.0	1.08	-146	
F2	12500		-135.0	20.0	1.08	-126	
F3	12501		-135.0	20.0	1.08	-129	
G1	12502	Fitzgerald Site (ElNp - 8), Besant, 1550 BP	-135.0	20.0	1.08	-107	
G2	12503		-135.0	20.0	1.08	-124	
G3	12504		-135.0	20.0	1.08	-107	
G4	12505		-135.0	20.0	1.08	-258	
G5	12506		-135.0	20.0	1.08	-81	
G6	12507		-135.0	20.0	1.08	-150	
G7	12508		-135.0	20.0	1.08	-124	
G8	12509		-135.0	20.0	1.08	-124	
G9	12510		-135.0	20.0	1.08	-226	
G10	12511		-135.0	20.0	1.08	-110	
G11	12512		-135.0	20.0	1.08	22	
G12	12513		-135.0	20.0	1.08	-114	
G13	12514		-135.0	20.0	1.08	-108	
G14	12515		-135.0	20.0	1.08	-110	
G15	12516		-135.0	20.0	1.08	-125	
H1	12517	Tschetter Site (FbNr - 1), Prairie Side Notched, 1050 BP	-135.0	20.0	1.08	-137	
H2	12518		-135.0	20.0	1.08	-126	
H3	12519		-135.0	20.0	1.08	-119	
H4	12520		-135.0	20.0	1.08	-112	
H5	12521		-135.0	20.0	1.08	-110	
H6	12522		-135.0	20.0	1.08	-119	
H7	12523		-135.0	20.0	1.08	-123	
H8	12524		-135.0	20.0	1.08	-112	
H9	12525		-135.0	20.0	1.08	-115	
H10	12526		-135.0	20.0	1.08	-193	
H11	12527		-135.0	20.0	1.08	-128	

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	δD (‰) Equilibration Waters (200 µl)	% Exchangeable Hydrogen	Fractionation Factor	δD (‰)	Comments
I1	*Values Are Aggregates	Oklahoma, Modern	-135.0	15.2	1.08	-62	
I2			-135.0	15.2	1.08	-59	
I3			-135.0	15.2	1.08	-80	
J1		Saskatoon, Modern	-135.0	20	1.08	-130	
J4			-135.0	20	1.08	-144	
J5		Yorkton, Modern	-135.0	20	1.08	-124	
J6			-135.0	20	1.08	-124	
Dietary Samples							
I4	*Values Are Aggregates	Oklahoma, Modern	-135.0	14.7	1.08	-103	Stomach Contents
J2		Saskatoon, Modern 2001	-135.0	17.5	1.08	-128	Feed
J3			-135.0	15.4	1.08	-184	Grass
J7		Saskatoon, Modern 2002	-135.0	19.6	1.08	-188	Grass
J8			-135.0	19	1.08	-156	Feed
J9		Yorkton, Modern 2002	-135.0	15.8	1.08	-215	Stomach Contents
J10			-135.0	18.7	1.08	-189	Grass

Table D6. Calculations For the Proportion of Exchangeable Hydrogen in Modern Oklahoma Bison Bone Collagen.

Samples	δD_{TA}	δD_{TB}	δD_{WA}	δD_{WB}	δD_n E = 60	δD_n E = 80	δD_n E = 100	*f@60*	*f@80*	*f@100*
I1/I2	-73.0	34.0	-135.0	525.0	**-71**	**-74**	**-77**	**0.15**	**0.15**	**0.15**
	-19.0	47.0	115.0	525.0	**-55**	**-58**	**-61**	**0.15**	**0.15**	**0.15**
	-70.0	-22.0	-135.0	115.0	**-67**	**-71**	**-75**	**0.18**	**0.18**	**0.17**
					-64	-68	-71	15.9		
					8	9	9	1.4		
I2/I3	-70.0	47.0	-135.0	525.0	**-67**	**-71**	**-74**	**0.17**	**0.16**	**0.16**
	-22.0	16.0	115.0	525.0	**-42**	**-43**	**-45**	**0.09**	**0.09**	**0.08**
	-87.0	-32.0	-135.0	115.0	**-88**	**-92**	**-97**	**0.21**	**0.20**	**0.20**
					-66	-69	-72	15.1		
					23	25	26	5.2		
I1/I3	-73.0	34.0	-135.0	525.0	**-71**	**-74**	**-77**	**0.15**	**0.15**	**0.15**
	-19.0	16.0	115.0	525.0	**-37**	**-38**	**-40**	**0.08**	**0.08**	**0.08**
	-87.0	-32.0	-135.0	115.0	**-88**	**-92**	**-97**	**0.21**	**0.20**	**0.20**
					-65	-68	-71	14.4		
					26	28	29	5.4		

Definitions

δD_{TA}	measured total A
δD_{TB}	measured total B
δD_{WA}	water A
δD_{WB}	water B
E = 60	fractionation factor
E = 80	fractionation factor
E = 100	fractionation factor
f	fraction of exchangeable
δD_n	non-exchangeable H (calculated)
Regression	% exchange using all data

Bison Bone Collagen

	Average	St.Dev
Oklahoma	15.2	4.3

Table D7. Calculations For the Proportion of Exchangeable Hydrogen in Modern Grass Samples.

Samples	δD_{TA}	δD_{TB}	δD_{WA}	δD_{WB}	δD_n E = 60	δD_n E = 80	δD_n E = 100	*f@60*	*f@80*	*f@100*
J3a	-178.0	-60.0	-135.0	525.0	**-197**	**-200**	**-203**	**0.17**	**0.17**	**0.16**
	-124.0	-51.0	115.0	525.0	**-186**	**-189**	**-192**	**0.17**	**0.16**	**0.16**
	-173.0	-130.0	-135.0	115.0	**-190**	**-193**	**-196**	**0.16**	**0.16**	**0.16**
					-191	-194	-197	16.3		
					6	6	6	0.4		
J3b	-173.0	-51.0	-135.0	525.0	**-192**	**-195**	**-198**	**0.17**	**0.17**	**0.17**
	-130.0	-60.0	115.0	525.0	**-190**	**-193**	**-195**	**0.16**	**0.16**	**0.16**
	-166.0	-131.0	-135.0	115.0	**-179**	**-181**	**-183**	**0.13**	**0.13**	**0.13**
					-187	-190	-192	15.3		
					7	8	8	1.9		
J3c	-178.0	-60.0	-135.0	525.0	**-197**	**-200**	**-203**	**0.17**	**0.17**	**0.16**
	-124.0	-60.0	115.0	525.0	**-177**	**-179**	**-182**	**0.15**	**0.14**	**0.14**
	-166.0	-131.0	-135.0	115.0	**-179**	**-181**	**-183**	**0.13**	**0.13**	**0.13**
					-184	-187	-189	14.7		
					11	12	12	1.6		
J7a	-180.0	-35.0	-135.0	525.0	**-205**	**-209**	**-213**	**0.21**	**0.20**	**0.20**
	-136.0	-41.0	115.0	525.0	**-225**	**-229**	**-233**	**0.22**	**0.21**	**0.21**
	-185.0	-124.0	-135.0	115.0	**-215**	**-220**	**-224**	**0.23**	**0.23**	**0.22**
					-215	-219	-223	21.5		
					10	10	10	1.0		
J7b	-185.0	-41.0	-135.0	525.0	**-211**	**-215**	**-219**	**0.21**	**0.20**	**0.20**
	-124.0	-49.0	115.0	525.0	**-188**	**-191**	**-194**	**0.17**	**0.17**	**0.17**
	-180.0	-133.0	-135.0	115.0	**-201**	**-204**	**-207**	**0.18**	**0.17**	**0.17**
					-200	-203	-207	18.2		
					12	12	12	1.6		

Definitions

δD_{TA}	measured total A
δD_{TB}	measured total B
δD_{WA}	water A
δD_{WB}	water B
E = 60	fractionation factor
E = 80	fractionation factor
E = 100	fractionation factor
f	fraction of exchangeable
δD_n	non-exchangeable H (calculated)
Regression	% exchange using all data

Grasses

	Average	St.Dev
Saskatoon A	15.4	1.5
Saskatoon B	19.6	1.9
Yorkton	18.7	1.4
Total	17.9	2.4

Samples	δD_{TA}	δD_{TB}	δD_{WA}	δD_{WB}	δD_n E = 60	δD_n E = 80	δD_n E = 100	*f@60*	*f@80*	*f@100*
J7c	-180.0	-35.0	-135.0	525.0	**-205**	**-209**	**-213**	**0.21**	**0.20**	**0.20**
	-136.0	-49.0	115.0	525.0	**-216**	**-219**	**-223**	**0.20**	**0.20**	**0.19**
	-180.0	-133.0	-135.0	115.0	**-201**	**-204**	**-207**	**0.18**	**0.17**	**0.17**
					-207	-211	-214	19.1		
					8	8	8	1.4		
J10a	-180.0	-48.0	-135.0	525.0	**-203**	**-206**	**-209**	**0.19**	**0.19**	**0.18**
	-116.0	-42.0	115.0	525.0	**-177**	**-180**	**-183**	**0.17**	**0.17**	**0.16**
	-182.0	-124.0	-135.0	115.0	**-210**	**-214**	**-218**	**0.22**	**0.21**	**0.21**
					-196	-200	-203	18.9		
					17	18	18	2.1		
J10b	-182.0	-42.0	-135.0	525.0	**-207**	**-210**	**-214**	**0.20**	**0.20**	**0.19**
	-124.0	-42.0	115.0	525.0	**-195**	**-199**	**-202**	**0.19**	**0.19**	**0.18**
	-181.0	-130.0	-135.0	115.0	**-204**	**-208**	**-211**	**0.19**	**0.19**	**0.19**
					-202	-206	-209	19.0		
					6	6	6	0.6		
J10c	-180.0	-48.0	-135.0	525.0	**-203**	**-206**	**-209**	**0.19**	**0.19**	**0.18**
	-116.0	-42.0	115.0	525.0	**-177**	**-180**	**-183**	**0.17**	**0.17**	**0.16**
	-181.0	-130.0	-135.0	115.0	**-204**	**-208**	**-211**	**0.19**	**0.19**	**0.19**
					-195	-198	-201	18.0		
					15	15	16	1.0		

Table D8. Calculations For the Proportion of Exchangeable Hydrogen in Modern Bison Feed Samples.

Samples	δD_{TA}	δD_{TB}	δD_{WA}	δD_{WB}	δD_n E = 60	δD_n E = 80	δD_n E = 100	*f@60*	*f@80*	*f@100*
J2a	-124.0	-3.0	-135.0	525.0	**-133**	**-136**	**-139**	**0.17**	**0.17**	**0.17**
	-81.0	-13.0	115.0	525.0	**-130**	**-133**	**-136**	**0.16**	**0.15**	**0.15**
	-125.0	-81.0	-135.0	115.0	**-133**	**-137**	**-140**	**0.17**	**0.16**	**0.16**
					-132	-135	-138	16.2		
					2	2	2	0.8		
J2b	-125.0	-3.0	-135.0	525.0	**-134**	**-137**	**-140**	**0.17**	**0.17**	**0.17**
	-81.0	-13.0	115.0	525.0	**-130**	**-133**	**-136**	**0.16**	**0.15**	**0.15**
	-141.0	-82.0	-135.0	115.0	**-158**	**-162**	**-166**	**0.22**	**0.22**	**0.21**
					-140	-144	-147	18.1		
					15	16	16	2.9		
J2c	-124.0	-3.0	-135.0	525.0	**-133**	**-136**	**-139**	**0.17**	**0.17**	**0.17**
	-81.0	-13.0	115.0	525.0	**-130**	**-133**	**-136**	**0.16**	**0.15**	**0.15**
	-141.0	-82.0	-135.0	115.0	**-158**	**-162**	**-166**	**0.22**	**0.22**	**0.21**
					-140	-144	-147	18.1		
					15	16	17	2.9		
J8a	-151.0	-17.0	-135.0	525.0	**-167**	**-171**	**-174**	**0.19**	**0.19**	**0.18**
	-92.0	-15.0	115.0	525.0	**-151**	**-154**	**-158**	**0.18**	**0.17**	**0.17**
	-159.0	-103.0	-135.0	115.0	**-179**	**-183**	**-187**	**0.21**	**0.21**	**0.20**
					-166	-169	-173	19.0		
					14	15	15	1.5		
J8b	-159.0	-15.0	-135.0	525.0	**-179**	**-183**	**-186**	**0.21**	**0.20**	**0.20**
	-103.0	-16.0	115.0	525.0	**-174**	**-178**	**-182**	**0.20**	**0.20**	**0.19**
	-154.0	-102.0	-135.0	115.0	**-171**	**-175**	**-179**	**0.20**	**0.19**	**0.19**
					-175	-179	-182	19.7		
					4	4	4	0.5		

Definitions

δD_{TA}	measured total A
δD_{TB}	measured total B
δD_{WA}	water A
δD_{WB}	water B
E = 60	fractionation factor
E = 80	fractionation factor
E = 100	fractionation factor
f	fraction of exchangeable
δD_n	non-exchangeable H (calculated)
Regression	% exchange using all data

Feed Supplement

	Average	St.Dev
Saskatoon A	17.5	2.5
Saskatoon B	19.0	1.2

Samples	δD_{TA}	δD_{TB}	δD_{WA}	δD_{WB}	δD_n E = 60	δD_n E = 80	δD_n E = 100	*f@60*	*f@80*	*f@100*
J8c	-151.0	-17.0	-135.0	525.0	**-167**	**-171**	**-174**	**0.19**	**0.19**	**0.18**
	-92.0	-16.0	115.0	525.0	**-150**	**-153**	**-157**	**0.17**	**0.17**	**0.17**
	-154.0	-102.0	-135.0	115.0	**-171**	**-175**	**-179**	**0.20**	**0.19**	**0.19**
					-163	-166	-170	18.4		
					11	11	12	1.0		

Table D9. Calculations For the Proportion of Exchangeable Hydrogen in Modern Bison Stomach Content Samples.

Samples	δD_{TA}	δD_{TB}	δD_{WA}	δD_{WB}	δD_n E = 60	δD_n E = 80	δD_n E = 100	*f@60*	*f@80*	*f@100*
I4a	-110.0	-1.0	-135.0	525.0	**-115**	**-118**	**-121**	**0.16**	**0.15**	**0.15**
	-69.0	-2.0	115.0	525.0	**-115**	**-118**	**-121**	**0.15**	**0.15**	**0.15**
	-102.0	-64.0	-135.0	115.0	**-105**	**-108**	**-111**	**0.14**	**0.14**	**0.14**
					-112	-115	-117	14.8		
					6	6	6	0.6		
I4b	-102.0	-2.0	-135.0	525.0	**-105**	**-108**	**-111**	**0.14**	**0.14**	**0.14**
	-64.0	0.0	115.0	525.0	**-106**	**-109**	**-112**	**0.15**	**0.14**	**0.14**
	-104.0	-66.0	-135.0	115.0	**-107**	**-110**	**-113**	**0.14**	**0.14**	**0.14**
					-106	-109	-112	14.2		
					1	1	1	0.3		
I4c	-110.0	-1.0	-135.0	525.0	**-115**	**-118**	**-121**	**0.16**	**0.15**	**0.15**
	-69.0	0.0	115.0	525.0	**-116**	**-119**	**-122**	**0.16**	**0.16**	**0.15**
	-104.0	-66.0	-135.0	115.0	**-107**	**-110**	**-113**	**0.14**	**0.14**	**0.14**
					-113	-116	-119	15.0		
					5	5	5	0.7		
J9a	-195.0	-84.0	-135.0	525.0	**-216**	**-219**	**-221**	**0.16**	**0.16**	**0.15**
	-154.0	-87.0	115.0	525.0	**-215**	**-218**	**-220**	**0.15**	**0.15**	**0.15**
	-197.0	-154.0	-135.0	115.0	**-219**	**-222**	**-225**	**0.16**	**0.16**	**0.16**
					-217	-220	-222	15.5		
					2	2	2	0.4		
J9b	-197.0	-87.0	-135.0	525.0	**-218**	**-221**	**-224**	**0.16**	**0.15**	**0.15**
	-154.0	-84.0	115.0	525.0	**-218**	**-221**	**-224**	**0.16**	**0.16**	**0.16**
	-201.0	-156.0	-135.0	115.0	**-225**	**-228**	**-231**	**0.17**	**0.17**	**0.16**
					-221	-223	-226	16.0		
					4	4	4	0.6		

Definitions

δD_{TA}	measured total A
δD_{TB}	measured total B
δD_{WA}	water A
δD_{WB}	water B
E = 60	fractionation factor
E = 80	fractionation factor
E = 100	fractionation factor
f	fraction of exchangeable
δD_n	non-exchangeable H (calculated)
Regression	% exchange using all data

Stomach Contents

	Average	St.Dev
Oklahoma	14.7	0.7
Yorkton	15.8	0.6

Samples	δD_{TA}	δD_{TB}	δD_{WA}	δD_{WB}	δD_n E = 60	δD_n E = 80	δD_n E = 100	*f@60*	*f@80*	*f@100*
J9c	-195.0	-84.0	-135.0	525.0	**-216**	**-219**	**-221**	**0.16**	**0.16**	**0.15**
	-154.0	-84.0	115.0	525.0	**-218**	**-221**	**-224**	**0.16**	**0.16**	**0.16**
	-201.0	-156.0	-135.0	115.0	**-225**	**-228**	**-231**	**0.17**	**0.17**	**0.16**
					-220	-223	-225	16.0		
					5	5	5	0.6		

Table D10. Un-corrected Hydrogen Isotope Composition of all Modern Samples Equilibrated with Multiple Waters of Varying δD Composition

Sample I1	δD of Equilibration Water						
	-135		115		525		
	Lab ID	δD	Lab ID	δD	Lab ID	δD	Oklahoma Bison Bone Collagen
	12810	-76	12811	-17	12999	34	
	13161	-72	12812	-24			
	13162	-71	13259	-16			
Mean		**-73**		-19		34	
Std. Dev.		3		4			

Sample I2	δD of Equilibration Water						
	-135		115		525		
	Lab ID	δD	Lab ID	δD	Lab ID	δD	Oklahoma Bison Bone Collagen
	12529	-72	12796	-24	12990	44	
	12794	-73	12798	-31	12991	49	
	12795	-65	13167	-12	12992	49	
Mean		**-70**		-22		47	
Std. Dev.		4		10		3	

Sample I3	δD of Equilibration Water						
	-135		115		525		
	Lab ID	δD	Lab ID	δD	Lab ID	δD	Oklahoma Bison Bone Collagen
	12530	-89	12802	-37	12993	19	
	12799	-84	12803	-40	12994	18	
	12800	-87	13168	-19	12995	12	
Mean		**-87**		-32		16	
Std. Dev.		3		11		4	

Sample I4	δD of Equilibration Water						
	-135		115		525		
	Lab ID	δD	Lab ID	δD	Lab ID	δD	Oklahoma Bison Stomach Contents
	12531	-110	12806	-69	12996	-1	
	12804	-102	12807	-64	12997	-2	
	12805	-104	12808	-66	12998	0	
Mean		**-105**		-66		-1	
Std. Dev.		4		3		1	

Sample J1	δD of Equilibration Water						
	-135		115		525		
	Lab ID	δD	Lab ID	δD	Lab ID	δD	Saskatoon Bison Bone Collagen 2001
	12528	-132	12792	-83	12987	-12	
	12789	-132	12793	-87	12989	-14	
	13160	-140	13258	-89			
Mean		**-135**		-86		-13	
Std. Dev.		5		3		1	

Sample J2	δD of Equilibration Water					
	-135		115		525	
	Lab ID	δD	Lab ID	δD	Lab ID	δD
	12814	-124	12816	-81	13003	-3
	12815	-125	12817	-81	13004	-13
	13163	-141	12818	-82		
Mean		**-130**		-81		-8
Std. Dev.		10		1		7

Saskatoon Feed Supplement 2001

Sample J3	δD of Equilibration Water					
	-135		115		525	
	Lab ID	δD	Lab ID	δD	Lab ID	δD
	12534	-178	13170	-124	13006	-60
	12820	-173	13171	-130	13178	-51
	13165	-166	13172	-131	13281	-60
Mean		**-172**		-128		-57
Std. Dev.		6		4		5

Saskatoon Grass Forage 2001

Sample J4	δD of Equilibration Water					
	-135		115		525	
	Lab ID	δD	Lab ID	δD	Lab ID	δD
	13179	-146	13182	-88	13186	10
	13180	-147	13183	-86	13252	18
	13181	-145	13184	-88	13262	11
Mean		**-146**		-87		13
Std. Dev.		1		1		4

Saskatoon Bison Bone Collagen 2001

Sample J5	δD of Equilibration Water					
	-135		115		525	
	Lab ID	δD	Lab ID	δD	Lab ID	δD
	13188	-129	13191	-77	13194	6
	13189	-131	13192	-88	13195	9
	13190	-130	13193	-72	13196	13
Mean		**-130**		-79		9
Std. Dev.		1		8		2

Yorkton Bison Bone Collagen

Sample J6	δD of Equilibration Water					
	-135		115		525	
	Lab ID	δD	Lab ID	δD	Lab ID	δD
	13197	-131	13200	-81	13203	4
	13199	-129	13201	-72	13253	28
	13248	-130	13202	-75	13254	19
Mean		**-130**		-76		17
Std. Dev.		1		5		12

Yorkton Bison Bone Collagen

Sample J7	δD of Equilibration Water					
	-135		115		525	
	Lab ID	δD	Lab ID	δD	Lab ID	δD
	13206	-180	13210	-136	13212	-35
	13207	-185	13211	-124	13214	-41
	13249	-180	13280	-133	13255	-49
Mean		**-182**		-131		-42
Std. Dev.		3		6		7

Saskatoon Grass Forage 2002

Sample J8	δD of Equilibration Water					
	-135		115		525	
	Lab ID	δD	Lab ID	δD	Lab ID	δD
	13215	-151	13218	-92	13221	-17
	13216	-159	13219	-103	13222	-15
	13217	-154	13220	-102	13223	-16
Mean		**-155**		-99		-16
Std. Dev.		4		6		1

Saskatoon Feed Supplement 2002

Sample J9	δD of Equilibration Water					
	-135		115		525	
	Lab ID	δD	Lab ID	δD	Lab ID	δD
	13224	-195	13228	-154	13230	-84
	13225	-197	13229	-154	13231	-87
	13226	-201	13264	-156	13232	-84
Mean		**-198**		-155		-85
Std. Dev.		3		1		2

Yorkton Bison Stomach Contents

Sample J10	δD of Equilibration Water					
	-135		115		525	
	Lab ID	δD	Lab ID	δD	Lab ID	δD
	13233	-180	13236	-116	13240	-48
	13250	-182	13237	-124	13241	-42
	13282	-181	13238	-130	13256	-42
Mean		**-181**		-123		-44
Std. Dev.		1		7		3

Yorkton Grass Forage

Appendix E. SAMPLE QUALITY AND SELECTION

E.1 Introduction

Stable isotope analysis of archaeological bone is dependent upon the state of tissue preservation. Several different methods have been proposed to assess sample integrity. The data upon which many of these are based, occurs as a by-product of mass spectrometry and any associated preparation of samples. The measurable amount of collagen in a sample and the subsequent elemental composition of that collagen provides insight into its integrity and thus, the reliability of isotopic data derived from it. Generally, techniques that assess the qualitative preservation of sample collagen are preferred to those that provide quantitative estimates. The techniques of collagen assessment that were utilized to scrutinize and select samples for this project are described in the following section. Where necessary, other data derived from sample preparation, combustion and analysis are also briefly discussed. Samples from two of the sites examined in this study exhibited collagen concentrations that were low enough to be of concern. A brief analysis of this problem and the rationale for inclusion of these samples in the overall analysis is subsequently provided.

E.2 Collagen Yields

The calculation of collagen yields is the simplest method through which to determine collagen integrity. Dry, modern bone can be over 25% collagen by weight (Schoeninger *et al.* 1989). Archaeological samples that yield concentrations between 5 and 25% are usually considered to have yielded sufficient collagen for isotopic analysis. Yields below 5% have been found to be associated with aberrant results. The reasons for this are not completely understood, however, it has been noted that non-collagenous organic residues may comprise a small portion of the organic fraction of bone. Thus, at low concentrations, these residues or extraneous contaminants may obscure collagen isotope ratios (Schoeninger *et al.* 1989). Collagen yield is an indicator of collagen

quantity, not quality. Some specimens have yielded large quantities of poorly preserved collagen, whereas others have yielded small amounts that produced good isotopic results (Pfeiffer and Varney 2000). The amount of collagen extracted from a sample may also be affected by the severity of the extraction technique. Each different method has advantages and risks that must be fully researched prior to application (See Moore 1989 for review).

Calculation of accurate collagen yields requires the assumption that the end product of collagen extraction and preparation is purified collagen protein. The extraction and purification methods utilized in this thesis follow the technique developed by Sealy and van der Merwe (1986) (See Appendix D). This extraction is considered to be harsh, relatively speaking, and usually results in the removal of inorganic and non-collagenous materials at the potential cost of reduced collagen yields (Moore 1989). Nevertheless, this technique was chosen because of the author's familiarity with it, and because loss of material via processing was not a concern due to the large quantities of bone available from each specimen. Various mass measurements were recorded for each sample in this study throughout the processing period. These measurements included mass of the unprocessed (original) sample, mass of the processed sample including the mass of its container, and mass of the empty container. With these measurements it was possible to calculate the collagen yield of each sample using the following formula:

$$\frac{[(\text{Container} + \text{Processed Sample Mass}) - (\text{Empty Container Mass})]}{\text{Unprocessed Sample Mass}} \times 100\% = \%\text{Yield}$$

This calculation was performed for every sample utilized in this study and the results are presented in Table E1.

Table E1. Calculated Collagen Yields For All Bison Bone Samples.

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	Original Sample Mass (g)	Processed Sample + Vial Mass (g)	Empty Vial Mass (g)	Processed Sample Mass (g)	Collagen Yield (%)
A1	12450	Heron Eden Site (EeOi-11), Cody Complex, 9150 BP	3.2503	13.34	13.0379	0.3021	**9.29**
A2	12451		3.1841	13.2632	13.0981	0.1651	**5.19**
A3	12452		3.3478	13.5917	13.1279	0.4638	**13.85**
A4	12453		3.146	13.6568	13.1613	0.4955	**15.75**
A5	12454		3.0301	13.5334	13.1734	0.36	**11.88**
A6	12455		3.0432	13.2414	13.108	0.1334	**4.38**
A7	12456		3.5061	13.5857	13.1621	0.4236	**12.08**
A8	12457		3.0533	13.2166	13.0621	0.1545	**5.06**
A9	12458		3.3065	13.268	13.0733	0.1947	**5.89**
A10	12459		3.0334	13.2602	13.1076	0.1526	**5.03**
A11	12460		3.2473	13.3048	13.1214	0.1834	**5.65**
A12	12461		3.5036	13.654	13.1457	0.5083	**14.51**
C1	12468	Norby Site (FaNq - 56), Mummy Cave, 7050 BP	3.5714	13.5296	13.0753	0.4543	**12.72**
C2	12469		3.2154	13.353	13.198	0.155	**4.82**
C3	12470		3.1464	13.3148	13.1605	0.1543	**4.90**
C4	12471		3.3004	13.1709	13.0542	0.1167	**3.54**
C5	12472		3.7334	13.2496	13.1641	0.0855	**2.29**
C6	12473		3.4919	13.3241	13.1271	0.197	**5.64**
C7	12474		3.4678	13.6227	13.2486	0.3741	**10.79**
C8	12475		3.08	13.3846	13.1133	0.2713	**8.81**
C9	12476		3.4422	13.3078	13.071	0.2368	**6.88**
C10	12477		3.3129	13.3614	13.153	0.2084	**6.29**
C11	12478		3.5345	13.388	13.0875	0.3005	**8.50**
C12	12479		3.0501	13.2655	13.2033	0.0622	**2.04**
C13	12480		3.4117	13.1965	13.1495	0.047	**1.38**
C14	12481		3.3603	13.2023	13.1495	0.0528	**1.57**
B1	12462	Gowen Sites (FaNq - 32), MummyCave, 5850 BP	3.2819	13.6213	13.2154	0.4059	**12.37**
B2	12463		3.1066	13.2869	13.1018	0.1851	**5.96**
B3	12464		3.2903	13.4675	13.1436	0.3239	**9.84**
B4	12465		3.4082	13.3246	13.0893	0.2353	**6.90**
B5	12466		3.0957	13.3618	13.1848	0.177	**5.72**
B6	12467		3.0819	13.3297	13.1523	0.1774	**5.76**
D1	12482	Amisk (FbNp - 17), Oxbow, 4350 BP	3.1557	13.4667	13.0888	0.3779	**11.98**
D2	12483		3.047	13.337	13.094	0.243	**7.98**
E1	12484	ɔw, 4200	3.3423	13.1461	13.0298	0.1163	**3.48**
E2	12485		3.501	13.2636	13.1772	0.0864	**2.47**
E3	12486		3.2292	13.2314	13.1001	0.1313	**4.07**
E4	12487		3.2176	13.0601	13.0286	0.0315	**0.98**

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	Original Sample Mass (g)	Processed Sample + Vial Mass (g)	Empty Vial Mass (g)	Processed Sample Mass (g)	Collagen Yield (%)
E5	12488	Harder Site (FbNs - 1), Oxb BP	3.1057	13.1761	13.1473	0.0288	**0.93**
E6	12489		3.6624	13.3285	13.2077	0.1208	**3.30**
E7	12490		3.3387	13.2412	13.1225	0.1187	**3.56**
E8	12491		3.0705	13.228	13.1481	0.0799	**2.60**
E9	12492		3.5328	13.2077	13.1088	0.0989	**2.80**
E10	12493		3.2502	13.1128	13.0907	0.0221	**0.68**
E11	12494		3.3588	13.1375	13.1028	0.0347	**1.03**
E12	12495		3.5608	13.2334	13.1116	0.1218	**3.42**
E13	12496		3.3484	13.2239	13.1633	0.0606	**1.81**
E14	12497		3.5059	13.2642	13.1765	0.0877	**2.50**
E15	12498		3.7775	13.1887	13.0855	0.1032	**2.73**
F1	12499	Thundercloud Site	3.144	13.3977	13.0541	0.3436	**10.93**
F2	12500	(FbNp - 25),	3.1467	13.4032	13.1491	0.2541	**8.08**
F3	12501	McKean, 3400 BP	3.0254	13.384	13.1747	0.2093	**6.92**
G1	12502	Fitzgerald Site (ElNp - 8), Besant, 1550 BP	3.1839	13.4458	13.101	0.3448	**10.83**
G2	12503		3.0497	14.0384	13.48	0.5584	**18.31**
G3	12504		3.3472	13.7084	13.2311	0.4773	**14.26**
G4	12505		3.8222	13.2965	13.1223	0.1742	**4.56**
G5	12506		3.2136	13.4074	13.1471	0.2603	**8.10**
G6	12507		3.4578	13.5048	13.1798	0.325	**9.40**
G7	12508		3.296	13.3893	13.1583	0.231	**7.01**
G8	12509		3.3006	13.45	13.2048	0.2452	**7.43**
G9	12510		3.3582	13.4398	13.115	0.3248	**9.67**
G10	12511		3.5773	13.4505	13.1043	0.3462	**9.68**
G11	12512		3.7724	13.4577	13.0603	0.3974	**10.53**
G12	12513		3.5994	13.5429	13.1889	0.354	**9.83**
G13	12514		3.2234	13.5098	13.0872	0.4226	**13.11**
G14	12515		3.7014	13.6518	13.1591	0.4927	**13.31**
G15	12516		3.1047	13.5448	13.1962	0.3486	**11.23**
H1	12517	Tschetter Site (FbNr - 1), Prairie Side Notched, 1050 BP	3.1541	13.5971	13.0815	0.5156	**16.35**
H2	12518		3.0922	13.6136	13.1563	0.4573	**14.79**
H3	12519		3.0971	13.475	13.0802	0.3948	**12.75**
H4	12520		3.2577	13.4789	13.153	0.3259	**10.00**
H5	12521		3.1599	13.4149	13.1469	0.268	**8.48**
H6	12522		3.0131	13.5518	13.2159	0.3359	**11.15**
H7	12523		3.1464	13.4149	13.1492	0.2657	**8.44**
H8	12524		3.0159	13.4129	13.1562	0.2567	**8.51**
H9	12525		3.203	13.8183	13.1323	0.686	**21.42**
H10	12526		3.2409	13.5132	13.067	0.4462	**13.77**
H11	12527		3.0464	13.3929	13.1217	0.2712	**8.90**
I1			3.38635	13.8479	13.1596	0.6883	**20.33**
I2	re s	Oklahoma, Modern	3.40006	13.957	13.13485	0.82215	**24.18**

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	Original Sample Mass (g)	Processed Sample + Vial Mass (g)	Empty Vial Mass (g)	Processed Sample Mass (g)	Collagen Yield (%)
I3	*Values A Aggregate		3.80618	14.1905	13.12829	1.06221	**27.91**
J1		Saskatoon, Modern	3.298	13.8147	13.09212	0.72258	**21.91**
J4			3.4755	13.9116	13.1795	0.7321	**21.06**
J5		Yorkton, Modern	3.4279	13.8212	13.0851	0.7361	**21.47**
J6			3.8357	14.0173	13.0939	0.9234	**24.07**

E.3 Carbon and Nitrogen Elemental Concentrations

Along with uncorrected isotopic ratios, mass spectrometry of prepared collagen samples produces data regarding the elemental composition of each sample. Measuring the absolute and relative proportions of these elements within collagen samples and then comparing them to those that are characteristic for the protein, may be a useful means towards the assessment of sample quality. Modern animals have carbon and nitrogen concentrations in bone collagen of roughly 15 - 47% and 5 - 17% by weight, respectively (Ambrose 1990, Varney 1994). Well preserved prehistoric bone collagen usually has more than 3% carbon and 1% nitrogen by weight (Ambrose 1993). It has thus, been suggested that samples that appear to have lower concentrations than these, should be excluded from consideration during any isotopic analysis (Ambrose 1993, Pfeiffer and Varney 2000). Unfortunately, a fair degree of variability in terms of "acceptable" results has also been noted (Ambrose 1990). Carbon and nitrogen concentrations should thus, never be used as a stand alone criteria. Additionally, the strength of acid used to demineralize the collagen during preparation may also influence the concentration of carbon and nitrogen. Use of less concentrated acids will help to mediate artificial reductions in concentration.

E.4 Atomic Carbon:Nitrogen (C/N) Ratios

The relative proportion of carbon to nitrogen in the collagen protein is also considered to be a diagnostic criterion. The end product of the demineralization and purification of bone samples for stable isotope analysis is theoretically pure collagen. Thus, the C/N ratio of each sample should reflect that of the collagen protein. The atomic ratio of carbon to nitrogen in unaltered collagen is roughly 3 (2.9 - 3.6) to 1 (DeNiro 1985). Deviance from within this range may indicate that a sample has been contaminated or improperly prepared. Higher than normal C/N ratios may indicate contamination from carbon-rich, nitrogen-poor sources such as soil humic acids, lipids, carbohydrates and carbonates; while low C/N ratios may indicate the presence of ammonia or small amines (Ambrose 1993, Varney 1994). The usefulness of C/N ratios to assess collagen quality have withstood some debate. Specimens that have undergone subtle modification may not be readily identified through this criterion alone (DeNiro

and Weiner 1988, Schoeninger *et al.* 1989). Some variability has been noted amongst samples with low collagen yields (Ambrose 1990). Nevertheless, C/N ratios may be a good general indicator of collagen preservation in situations where contamination is thought to be of low risk and with samples that yield adequate amounts of collagen (Bocherens 2002, Personal Communication).

The carbon and nitrogen elemental compositions that are obtained from mass spectrometry of samples are in a raw, un-standardized form. These percentages in fact represent relative mass and not atomic mass composition. The carbon/nitrogen ratio is simply calculated by dividing the former mass by the latter. As each sample is tested multiple times, means for the resultant mass ratios must be determined for both carbon and nitrogen. A correction calculation is then needed to standardize the data, converting it into atomic ratios. This is accomplished by multiplying the un-corrected mass ratios by a predetermined standard correction factor of 1.167 g/mole (the ratio of the atomic molar mass of nitrogen to carbon, i.e. 14.01/12.01). Once this has been completed, atomic C/N ratios must be determined for each sample. The resultant values become the reported C/N ratios for each sample.

Table E2 presents both the carbon and nitrogen concentrations, as well as the C/N ratios that were determined for each specimen in this study. This information is not reported for specimens E1, E8, E15, J4, J5 and J6. Equipment failure during the experimental runs of these samples prevented the measurement of these data. The loss of this information is not significant in terms of the three modern samples. There is no concern over the preservation of collagen in these instances. The loss of these data for the three Harder site specimens is unfortunate for reasons explained later.

Table E2. Carbon and Nitrogen Concentrations and Calculated Carbon to Nitrogen (C/N) Ratios For All Bison Bone Samples

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	EA Sample Mass (mg)	Elemental % Composition (C)	Elemental % Composition (N)	C/N Elemental Mass Ratio	C/N Atomic Mass Ratio
A1	12450	Heron Eden Site (EeOi-11), Cody Complex, 9150 BP	1.404	42.0	15.3	2.7	**3.2**
A2	12451		1.411	43.8	15.9	2.8	**3.2**
A3	12452		1.456	10.7	3.1	3.5	**4.1**
A4	12453		1.583	7.2	1.8	3.9	**4.6**
A5	12454		1.515	42.8	15.7	2.7	**3.2**
A6	12455		1.445	30.5	10.7	2.8	**3.3**
A7	12456		1.588	6.0	1.6	3.7	**4.4**
A8	12457		1.592	28.6	10.1	2.8	**3.3**
A9	12458		1.491	40.2	14.5	2.8	**3.2**
A10	12459		1.496	30.9	11.0	2.8	**3.3**
A11	12460		1.517	32.2	11.5	2.8	**3.3**
A12	12461		1.523	4.3	1.4	3.1	**3.7**
C1	12468	Norby Site (FaNq - 56), Mummy Cave, 7050 BP	1.429	12.2	3.0	4.0	**4.7**
C2	12469		1.547	4.6	1.0	4.4	**5.2**
C3	12470		1.472	9.5	2.7	3.5	**4.1**
C4	12471		1.406	17.1	5.5	3.1	**3.6**
C5	12472		1.528	19.5	6.2	3.1	**3.7**
C6	12473		1.506	6.6	1.9	3.5	**4.0**
C7	12474		1.593	17.8	6.0	3.0	**3.5**
C8	12475		1.585	4.4	1.1	4.0	**4.6**
C9	12476		1.494	5.5	1.4	3.8	**4.4**
C10	12477		1.548	6.0	1.6	3.6	**4.2**
C11	12478		1.407	12.3	3.9	3.2	**3.7**
C12	12479		1.497	38.8	12.9	3.0	**3.5**
C13	12480		1.553	38.8	13.0	3.0	**3.5**
C14	12481		1.432	40.5	13.5	3.0	**3.5**
B1	12462	Gowen Sites (FaNq - 32), MummyCave, 5850 BP	1.511	13.5	4.3	3.1	**3.6**
B2	12463		1.545	23.0	7.9	2.9	**3.4**
B3	12464		1.575	24.7	8.3	3.0	**3.5**
B4	12465		1.449	38.9	13.5	2.9	**3.4**
B5	12466		1.557	27.2	9.2	3.0	**3.5**
B6	12467		1.578	27.9	9.3	3.0	**3.5**
D1	12482	Amisk (FbNp - 17), Oxbow, 4350 BP	1.419	39.3	14.8	2.7	**3.1**
D2	12483		1.529	42.6	15.3	2.8	**3.3**
E1	12484	ow, 4200	* No Values Available Due to Equipment Failure				
E2	12485		1.558	13.4	3.8	3.5	**4.1**
E3	12486		1.450	9.4	2.8	3.4	**4.0**
E4	12487		1.585	35.0	12.5	2.8	**3.3**

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	EA Sample Mass (mg)	Elemental % Composition (C)	Elemental % Composition (N)	C/N Elemental Mass Ratio	C/N Atomic Mass Ratio
E5	12488	Harder Site (FbNs - 1), Oxb BP	1.484	35.1	11.9	3.0	**3.4**
E6	12489		1.525	28.6	9.4	3.0	**3.5**
E7	12490		1.462	38.5	12.9	3.0	**3.5**
E8	12491		* No Values Available Due to Equipment Failure				
E9	12492		1.575	8.1	2.2	3.8	**4.4**
E10	12493		1.443	30.7	11.0	2.8	**3.3**
E11	12494		1.497	28.2	8.9	3.2	**3.7**
E12	12495		1.440	38.6	13.0	3.0	**3.5**
E13	12496		1.521	7.3	1.9	3.8	**4.4**
E14	12497		1.443	19.4	6.5	3.0	**3.5**
E15	12498		* No Values Available Due to Equipment Failure				
F1	12499	Thundercloud Site (FbNp - 25), McKean, 3400 BP	1.597	41.7	15.1	2.8	**3.2**
F2	12500		1.417	41.4	14.3	2.9	**3.4**
F3	12501		1.444	42.2	14.4	2.9	**3.4**
G1	12502	Fitzgerald Site (ElNp - 8), Besant, 1550 BP	1.505	45.5	16.4	2.8	**3.2**
G2	12503		1.542	46.6	17.0	2.7	**3.2**
G3	12504		1.488	44.9	16.3	2.8	**3.2**
G4	12505		1.543	44.8	14.7	3.1	**3.6**
G5	12506		1.492	45.6	16.1	2.8	**3.3**
G6	12507		1.522	46.2	16.3	2.8	**3.3**
G7	12508		1.505	42.6	14.8	2.9	**3.4**
G8	12509		1.550	45.0	15.9	2.8	**3.3**
G9	12510		1.545	44.2	15.8	2.8	**3.3**
G10	12511		1.472	45.1	16.2	2.8	**3.2**
G11	12512		1.540	45.3	16.6	2.7	**3.2**
G12	12513		1.497	45.5	15.9	2.9	**3.3**
G13	12514		1.537	45.9	16.7	2.7	**3.2**
G14	12515		1.499	48.7	17.5	2.8	**3.2**
G15	12516		1.552	46.7	16.8	2.8	**3.2**
H1	12517	Tschetter Site (FbNr - 1), Prairie Side Notched, 1050 BP	1.530	28.6	10.5	2.7	**3.2**
H2	12518		1.502	42.8	15.5	2.8	**3.2**
H3	12519		1.536	36.8	13.8	2.7	**3.1**
H4	12520		1.578	40.5	14.6	2.8	**3.2**
H5	12521		1.527	43.4	15.3	2.8	**3.3**
H6	12522		1.542	44.1	15.4	2.9	**3.3**
H7	12523		1.521	42.7	14.6	2.9	**3.4**
H8	12524		1.476	42.7	15.6	2.7	**3.2**
H9	12525		1.443	30.5	11.6	2.6	**3.1**
H10	12526		1.427	45.6	16.1	2.8	**3.3**
H11	12527		1.558	43.6	15.4	2.8	**3.3**
I1	s Are lates	Oklahoma, Modern	1.547	47.9	17.1	2.8	**3.3**
I2			1.490	30.4	10.8	2.8	**3.3**
I3			1.521	50.9	16.2	3.1	**3.7**

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	EA Sample Mass (mg)	Elemental % Composition (C)	Elemental % Composition (N)	C/N Elemental Mass Ratio	C/N Atomic Mass Ratio
J1	*Values Aggreg	Saskatoon, Modern	1.448	49.7	18.0	2.8	3.2
J4			* No Values Available Due to Equipment Failure				
J5		Yorkton, Modern					
J6							

E.5 Sample Combustion and Gas Volumes

Each specimen examined in this study was prepared offline prior to the determination of D/H ratios. Data concerning the amounts of specific gases produced during combustion were obtained from each sample during the gas separation phase and after mass spectrometry had taken place. Although these specific gas volumes do not directly characterize collagen quality, they can be used as indicators of potential problems with a particular specimen. Aberrations in sample gas volumes may be suggestive of problems with sample preparation or purification, equipment failure, or in certain instances even sample quality problems. Because gas volumes are usually measured in terms of pressure, only those samples that are separated on the same line or injected into the same instrument can be compared. This is due to the fact that pressure measurements are determined through a combination of the actual gas volume, as measured within a vessel of a specific volume.

CO_2, N_2 and H_2O volumes were obtained during the preparation and analysis of each specimen used in this thesis. These volumes were measured in millibarrs of pressure. Generally speaking, 7.5 mg of purified collagen was expected to produce between 200 and 400 mb of CO_2, 10 - 25 mb of N_2 and 30 - 70 mb of H_2O. Deviation from these ranges in terms of absolute volume, as well as changes in the relative proportion of one gas to another, may be used to discriminate samples. Unfortunately, the collagen yields of the samples must also be taken into account during evaluation. Samples with generally low collagen yields may be expected to produce less gas upon combustion. Especially if a percentage of the sample itself is non-collagenous, non-combustible material. Problems with both the Harder and Norby site materials resulted in such circumstances and are described later. Gas volume data for each sample prepared offline for δD analysis, is presented in Table E3.

This gas volume data for all of the modern specimens represent aggregate values. As a result of the static equilibrations used to determine the proportion of exchangeable hydrogen in each of the modern collagen samples, several trials were made of each individual specimen. Therefore, the reported gas volumes for each modern sample are an estimated mean based upon the results of multiple trials.

Table E3. Measured Gas Volumes From Combusted Samples Prepared For δD Analysis.

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	Sample Weight (mg)	δD (‰) Equilibration Water (200 μm)	Approximate N_2 Gas Volume (mb)	Approximate CO_2 Gas Volume (mb)	Approximate H_2O Vapour Volume (mb)	Comments
A1	12450	Heron Eden Site (EeOi-11), Cody Complex, 9150 BP	7.7	-135	18	328	0	Bad Trace
A2	12451		7.7	-135	16	319	57	
A3	12452		7.5	-135	0	47	1	Low Nitrogen
A4	12453		7.5	-135	0	53	4	Low Nitrogen
A5	12454		7.3	-135	16	314	59	
A6	12455		7.4	-135	11	245	46	
A7	12456		7.3	-135	0	38	2	Low Nitrogen
A8	12457		7.5	-135	10	206	36	
A9	12458		7.5	-135	16	329	58	
A10	12459		7.5	-135	12	245	49	
A11	12460		7.4	-135	12	195	35	
A12	12461		7.9	-135	0	48	51	
C1	12468	ite (FaNq - 56), Mummy Cave, 7050 BP	7.6	-135	1	70	72	
C2	12469		7.6	-135	2	76	82	
C3	12470		7.8	-135	0	34	22	
C4	12471		7.6	-135	3	85	12	
C5	12472		7.7	-135	7	182	34	
C6	12473		7.7	-135	0	39	23	Un-balanced Ion Gauge
C7	12474		7.8	-135	4	109	14	
C8	12475		7.7	-135	0	27	83	Un-balanced Ion Gauge
C9	12476		7.6	-135	0	23	28	Un-balanced Ion Gauge
C10	12477		7.4	-135	2	83	11	

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	Sample Weight (mg)	δD (‰) Equilibration Water (200 μm)	Approximate N_2 Gas Volume (mb)	Approximate CO_2 Gas Volume (mb)	Approximate H_2O Vapour Volume (mb)	Comments
C11	12478	Norby S	7.5	-135	2	80	11	
C12	12479		7.5	-135	13	275	48	
C13	12480		7.6	-135	14	292	53	
C14	12481		7.6	-135	15	305	59	
B1	12462	Gowen Sites (FaNq - 32), MummyCave, 5850 BP	7.3	-135	3	109	16	
B2	12463		7.6	-135	8	184	30	
B3	12464		7.4	-135	5	141	24	
B4	12465		7.7	-135	15	294	47	
B5	12466		7.3	-135	10	240	43	
B6	12467		7.8	-135	4	124	21	
D1	12482	Amisk (FbNp - 17), Oxbow, 4350 BP	7.8	-135	16	298	58	
D2	12483		7.5	-135	15	286	53	
E1	12484	Harder Site (FbNs - 1), Oxbow, 4200 BP	7.4	-135	16	303	57	
E2	12485		7.4	-135	1	82	86	
E3	12486		7.4	-135	1	79	11	
E4	12487		7.5	-135	13	266	47	
E5	12488		7.6	-135	13	272	50	
E6	12489		7.8	-135	10	202	36	
E7	12490		7.9	-135	14	278	52	
E8	12491		7.8	-135	14	301	51	
E9	12492		7.9	-135	11	89	10	
E10	12493		7.6	-135	12	203	\	
E11	12494		7.9	-135	10	210	35	
E12	12495		7.5	-135	15	288	54	
E13	12496		7.6	-135	0	40	42	
E14	12497		7.7	-135	7	101	30	

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	Sample Weight (mg)	δD (‰) Equilibration Water (200 μm)	Approximate N_2 Gas Volume (mb)	Approximate CO_2 Gas Volume (mb)	Approximate H_2O Vapour Volume (mb)	Comments
E15	12498		7.5	-135	0	81	11	
F1	12499	Thundercloud Site	7.7	-135	17	327	64	
F2	12500	(FbNp - 25),	7.5	-135	10	136	19	
F3	12501	McKean, 3400 BP	7.4	-135	13	307	55	
G1	12502		7.5	-135	16	324	60	
G2	12503		7.7	-135	18	339	66	
G3	12504		7.6	-135	17	369	61	
G4	12505		7.5	-135	16	309	33	Questionable H-values
G5	12506		7.8	-135	15	330	96	Questionable H-values
G6	12507		7.7	-135	16	331	25	Questionable H-values
G7	12508		7.6	-135	16	313	57	
G8	12509		7.8	-135	17	332	65	
G9	12510		7.6	-135	15	304	47	Un-balanced Ion Gauge
G10	12511		7.6	-135	17	322	60	
G11	12512		7.8	-135	18	329	62	Questionable H-values
G12	12513		7.3	-135	16	312	57	
G13	12514		7.3	-135	17	321	62	
G14	12515		7.7	-135	18	348	64	
G15	12516		7.7	-135	17	327	59	
H1	12517		7.6	-135	17	323	62	
H2	12518		7.4	-135	17	313	60	
H3	12519		7.8	-135	16	340	63	
H4	12520		7.3	-135	17	315	60	
H5	12521		7.4	-135	16	306	54	
H6	12522		7.5	-135	17	317	62	
H7	12523		7.5	-135	17	323	59	

Fitzgerald Site (ElNp - 8), Besant, 1550 BP

ar Site (FbNr - 1), de Notched, 1050 BP

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	Sample Weight (mg)	δD (‰) Equilibration Water (200 μm)	Approximate N_2 Gas Volume (mb)	Approximate CO_2 Gas Volume (mb)	Approximate H_2O Vapour Volume (mb)	Comments
H8	12524	Tschette Prairie Si	7.7	-135	18	335	65	
H9	12525		7.7	-135	17	338	68	RadioCarbon Date Prob.
H10	12526		7.6	-135	17	352	57	Questionable H-values
H11	12527		7.4	-135	16	302	58	These values all represent aggregates averaged from several trials of each specimen.
I1	*Values Are Aggregates	Oklahoma, Modern	7.5	-135	16	333	59	
I2			7.5	-135	16	333	63	
I3			7.5	-135	15	355	77	
J1		Saskatoon, Modern	7.5	-135	15	328	59	
J4			7.5	-135	17	320	61	
J5		Yorkton, Modern	7.5	-135	18	320	60	
J6			7.5	-135	18	300	57	

E.6 Duration of Collagen Extractions (Demineralization)

As previously noted, the collagen extraction technique utilized in this study relies upon the visual examination of the sample and other subjective criteria for the determination of demineralization completion. Because demineralization should not result in a substantial reduction of collagen quantity, no correlation between the duration of demineralization and collagen yield should be observed. No strong correlation between extraction duration and collagen yield was noted ($r^2 = 0.36$). Correlations with the duration of demineralization were also low for carbon concentration ($r^2 = 0.46$), nitrogen concentration ($r^2 = 0.48$) and for C/N ratio ($r^2 = 0.27$). Correlations between collagen yield and carbon concentration ($r^2 = 0.09$), nitrogen concentration ($r^2 = 0.1$) and C/N ratio ($r^2 = 0.05$), were also not significant (see Table E4).

E.7 Discussion of Norby and Harder Site Sample Collagen Yields

Despite the fact that the majority of Norby and Harder site samples provided "good quality" collagen as measured through C/N ratios and carbon and nitrogen concentrations, all of the samples from these two sites yielded low absolute collagen concentrations. Samples with low collagen yields (>5%) are usually excluded from studies involving stable isotope analysis because investigations have occasionally linked low yield specimens with aberrant isotopic results (Ambrose and DeNiro 1989, Schoeninger *et al.* 1989). Nevertheless, collagen yield is not a particularly good indicator of collagen quality and some archaeological samples do retain collagenous residues at yields as low as 0.8 - 4.0 % (Ambrose 1990, Ambrose and DeNiro 1989, DeNiro and Weiner 1988a).

In the case of both the Norby and Harder sample sets, the majority of specimens yielded C/N ratios and carbon and nitrogen concentrations that are considered to be indicative of unaltered collagen. The reduced yields in both instances have been traced to problems with the initial sample preparation that should theoretically have had no effect on the sample quality. During collagen extraction, the Norby bone samples liquefied as the demineralization progressed. In situations where overall collagen

concentrations are low, this reaction is not necessarily uncommon. However, in order to recover all collagenous material from solution, a filtration system is usually employed.

Unfortunately, during filtration of the Norby site samples used in this study, a small amount of collagenous material was lost in each case. This resulted from the selection of glass fiber filters that were too fine and did not allow for all of the moisture to pass through. Additionally, portions of the glass filters themselves were mixed in with the sample. Because collagen yield determinations are simply made through a mechanical measurement of the material resulting from the extraction procedure, the overall loss of material through filtration would act to artificially reduce the resulting yield measurements. The inclusion of glass filter fragments in the freeze-dried samples would further confuse the mass data used to calculate the collagen yields. Fortunately, the glass filter material is inert, and would therefore not produce gas during sample combustion. Thus, inclusion of the glass filter material would not act to contaminate the resultant isotopic ratios. It was subsequently determined that low collagen yields alone were not a sufficient reason to exclude the Norby site specimens from consideration. In instances where C/N ratios and carbon and nitrogen concentrations indicated the presence of good quality collagen, the decision was made to include these samples in the analysis.

Similar problems were encountered during the preparation and assessment of the Harder site specimens. During demineralization, it became apparent that the various samples contained sediment which had not been removed during pre-extraction cleansing. Prior to collagen extraction the various bone chunks were mechanically cleaned and hydrosonically bathed to remove burial debris. The use of the hydrosonic bath helps to dislodge and remove sediment from any cancellous portions of the bone that may remain after segmentation. After the inorganic structure of the Harder site bone samples had dissolved, it became apparent that sediment had been attached to the samples themselves and now occurred as a precipitate in the demineralizing solution.

The presence of a potential contaminant was disturbing considering that bones from the Harder site had undergone previous scrutiny from which potential qualitative alteration of the bone had been suggested (Dyck 1977, Morlan 1994). In addition to a "porcelain like appearance" much of the Harder site material contained $CaCO_3$

concretions. Nevertheless, C/N ratios and carbon and nitrogen concentrations from many of the prepared samples indicated the presence of qualitatively intact collagen and the use of HCl acid in the extraction phase should have reduced the $CaCO_3$ to solution, removing it from the samples. The sediment that remained as a precipitate during collagen extraction was then filtered from the samples. However, due to the same filtration problems that affected the Norby site samples, not all of the sediment could be removed.

If the sediment were found to be inorganic and inert, it would not affect the isotopic ratios of the purified collagen. Three samples of the Harder site bone were sent to the Department of Geology at the University of Saskatchewan to undergo X-ray diffraction analysis in an attempt to identify the contamination. X-rays are used as a means of identifying minerals by their structure (Press and Siever 1986). Essentially, the x-ray beams form a diffraction pattern based upon the angles at which they reflect from the planes within a crystal. The spacing and the intensity of the reflections depend on the kinds of atoms, as well as their arrangements. Thus, each mineral is distinctive (Press and Siever 1986).

The first of the three samples (E1) contained purified, freeze dried collagen that was qualitatively indistinguishable from the uncontaminated samples that were obtained from other sites used in this thesis. The remaining two samples (E8, E15) contained purified, freeze dried collagen that appeared to contain sediment contamination. The x-ray diffraction analysis, as interpreted by researchers in the Department of Geology, indicated the presence of quartz within sample E15. Sample E15 was notably the most contaminated in appearance and thus, the sediment present in other samples was assumed to be quartz silicates as well. All samples were also noted to contain unidentifiable organics, which were assumed to be collagen. The presence of quartz silica (i.e. sand) in both the burial environment and any subsequently exhumed materials is not unexpected. Quartz in the extracted collagen would not affect any resultant isotopic ratios as quartz would not combust at the temperatures utilized in this analysis.

The presence of quartz also helps to explain the low collagen yields associated with the Harder site bone samples. The masses of the unprocessed specimens would be

artificially inflated by the presence of sediment and $CaCO_3$. During collagen extraction, the $CaCO_3$ would solubilize and along with most of the sediment, be removed during filtration. The final mass of the specimen would thus, not simply reflect the demineralization and chemical purification of the sample, but also the loss of extraneous $CaCO_3$ and sediment. The final Harder specimen collagen yields are therefore, felt to be an artifact of the preparation methods, and not necessarily an indicator of diagenetically reduced collagen concentrations. Most of the Harder sample C/N ratio and carbon and nitrogen concentration data indicated the presence of unmodified collagen and thus, these samples were subsequently included in the analysis.

E.8 Selection of Usable Samples

Table E4 presents a summary of the determinants used to identify samples which were not appropriate for further analysis. Those specimens which are "highlighted" in the table were disregarded. The excluded samples may have failed to meet one or more of the criteria that were previously discussed in this appendix. For both the Norby and Harder site sample groups, the collagen yield data was not used during this evaluation for reasons which have also been previously detailed. Samples A1, E10, E14, G4, G6, G5, G9, G11 and H10 were excluded because they yielded highly suspect D/H ratios. These values were either unexplainably high or unexplainably low, suggesting potential contamination at some point during the analysis (see Appendix D). Many of these samples were also associated with aberrant gas volume yields. Samples A3, A4, A7, C1, C2, C3, C6, C8, C9, C10, E2, E3, E9 and E13 were all discredited as a result of demonstrating aberrant carbon and nitrogen concentrations and/or C/N ratios.

Sample A12 demonstrated gas volumes that were uncharacteristic for the Heron Eden sample group and that seemed low given the specimen's high collagen yield. Sample H9 was removed from consideration for reasons detailed in Appendix B. A radiocarbon date determined from this specimen was too recent to be acceptable and the collagen yield from this sample was uncharacteristic of the other Tschetter specimens. Finally, specimen E15 was excluded from further analysis because its appearance was

highly suspect. Without data concerning the elemental composition of the collagen extracted from this sample, there was no way to evaluate its quality. In the end, 26 of the 85 total specimens were considered to be unfit for further investigation. Thus, 59 specimens were used in the final analysis for this project (see Appendix F).

Table E4. Summary Table of Selection Criteria Indicating Those Samples Excluded From Analysis.

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	Approximate N_2 Gas Volume (mb)	Approximate CO_2 Gas Volume (mb)	Approximate H_2O Vapour Volume (mb)	Duration of Collagen Extraction (24 hr. periods)	Collagen Yield (%)	Elemental % Composition (C)	Elemental % Composition (N)	C:N Atomic Mass Ratio
A1	12450	Heron Eden Site (EeOi-11), Cody Complex, 9150 BP	18	328	0	26	9.29	42.0	15.3	3.2
A2	12451		16	319	57	14	5.19	43.8	15.9	3.2
A3	12452		0	47	1	10	13.85	10.7	3.1	4.1
A4	12453		0	53	4	10	15.75	7.2	1.8	4.6
A5	12454		16	314	59	26	11.88	42.8	15.7	3.2
A6	12455		11	245	46	12	4.38	30.5	10.7	3.3
A7	12456		0	38	2	10	12.08	6.0	1.6	4.4
A8	12457		10	206	36	12	5.06	28.6	10.1	3.3
A9	12458		16	329	58	22	5.89	40.2	14.5	3.2
A10	12459		12	245	49	12	5.03	30.9	11.0	3.3
A11	12460		12	195	35	12	5.65	32.2	11.5	3.3
A12	12461		0	48	51	10	14.51	4.3	1.4	3.7
C1	12468	ite (FaNq - 56), Mummy Cave, 7050 BP	1	70	72	10	12.72	12.2	3.0	4.7
C2	12469		2	76	82	10	4.82	4.6	1.0	5.2
C3	12470		0	34	22	10	4.90	9.5	2.7	4.1
C4	12471		3	85	12	16	3.54	17.1	5.5	3.6
C5	12472		7	182	34	10	2.29	19.5	6.2	3.7
C6	12473		0	39	23	12	5.64	6.6	1.9	4.0
C7	12474		4	109	14	10	10.79	17.8	6.0	3.5
C8	12475		0	27	83	10	8.81	4.4	1.1	4.6
C9	12476		0	23	28	10	6.88	5.5	1.4	4.4
C10	12477		2	83	11	10	6.29	6.0	1.6	4.2

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	Approximate N_2 Gas Volume (mb)	Approximate CO_2 Gas Volume (mb)	Approximate H_2O Vapour Volume (mb)	Duration of Collagen Extraction (24 hr. periods)	Collagen Yield (%)	Elemental % Composition (C)	Elemental % Composition (N)	C:N Atomic Mass Ratio
C11	12478	Norby S	2	80	11	15	8.50	12.3	3.9	3.7
C12	12479		13	275	48	10	2.04	38.8	12.9	3.5
C13	12480		14	292	53	10	1.38	38.8	13.0	3.5
C14	12481		15	305	59	10	1.57	40.5	13.5	3.5
B1	12462	Gowen Sites (FaNq - 32), MummyCave, 5850 BP	3	109	16	10	12.37	13.5	4.3	3.6
B2	12463		8	184	30	10	5.96	23.0	7.9	3.4
B3	12464		5	141	24	10	9.84	24.7	8.3	3.5
B4	12465		15	294	47	15	6.90	38.9	13.5	3.4
B5	12466		10	240	43	15	5.72	27.2	9.2	3.5
B6	12467		4	124	21	15	5.76	27.9	9.3	3.5
D1	12482	Amisk (FbNp - 17), Oxbow, 4350 BP	16	298	58	10	11.98	39.3	14.8	3.1
D2	12483		15	286	53	10	7.98	42.6	15.3	3.3
E1	12484	der Site (FbNs - 1), Oxbow, 4200 BP	16	303	57	15	3.48	* Equipment Failure		
E2	12485		1	82	86	15	2.47	13.4	3.8	4.1
E3	12486		1	79	11	15	4.07	9.4	2.8	4.0
E4	12487		13	266	47	15	0.98	35.0	12.5	3.3
E5	12488		13	272	50	15	0.93	35.1	11.9	3.4
E6	12489		10	202	36	15	3.30	28.6	9.4	3.5
E7	12490		14	278	52	15	3.56	38.5	12.9	3.5
E8	12491		14	301	51	15	2.60	* Equipment Failure		
E9	12492		11	89	10	15	2.80	8.1	2.2	4.4
E10	12493		12	203	\	15	0.68	30.7	11.0	3.3
E11	12494		10	210	35	15	1.03	28.2	8.9	3.7
E12	12495		15	288	54	15	3.42	38.6	13.0	3.5
E13	12496		0	40	42	15	1.81	7.3	1.9	4.4

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	Approximate N_2 Gas Volume (mb)	Approximate CO_2 Gas Volume (mb)	Approximate H_2O Vapour Volume (mb)	Duration of Collagen Extraction (24 hr. periods)	Collagen Yield (%)	Elemental % Composition (C)	Elemental % Composition (N)	C:N Atomic Mass Ratio
E14	12497	Har	7	101	30	15	2.50	19.4	6.5	3.5
E15	12498		0	81	11	15	2.73	* Equipment Failure		
F1	12499	Thundercloud Site	17	327	64	24	10.93	41.7	15.1	3.2
F2	12500	(FbNp - 25),	10	136	19	24	8.08	41.4	14.3	3.4
F3	12501	McKean, 3400 BP	13	307	55	16	6.92	42.2	14.4	3.4
G1	12502		16	324	60	40	10.83	45.5	16.4	3.2
G2	12503		18	339	66	54	18.31	46.6	17.0	3.2
G3	12504		17	369	61	40	14.26	44.9	16.3	3.2
G4	12505		16	309	33	26	4.56	44.8	14.7	3.6
G5	12506		15	330	96	28	8.10	45.6	16.1	3.3
G6	12507		16	331	25	28	9.40	46.2	16.3	3.3
G7	12508		16	313	57	28	7.01	42.6	14.8	3.4
G8	12509		17	332	65	26	7.43	45.0	15.9	3.3
G9	12510		15	304	47	28	9.67	44.2	15.8	3.3
G10	12511		17	322	60	28	9.68	45.1	16.2	3.2
G11	12512		18	329	62	28	10.53	45.3	16.6	3.2
G12	12513		16	312	57	28	9.83	45.5	15.9	3.3
G13	12514		17	321	62	28	13.11	45.9	16.7	3.2
G14	12515		18	348	64	40	13.31	48.7	17.5	3.2
G15	12516	Fitzgerald Site (ElNp - 8), Besant, 1550 BP	17	327	59	40	11.23	46.7	16.8	3.2
H1	12517		17	323	62	30	16.35	28.6	10.5	3.2
H2	12518		17	313	60	30	14.79	42.8	15.5	3.2
H3	12519		16	340	63	30	12.75	36.8	13.8	3.1
H4	12520		17	315	60	30	10.00	40.5	14.6	3.2
H5	12521	: (FbNr - 1), tched, 1050	16	306	54	26	8.48	43.4	15.3	3.3

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	Approximate N_2 Gas Volume (mb)	Approximate CO_2 Gas Volume (mb)	Approximate H_2O Vapour Volume (mb)	Duration of Collagen Extraction (24 hr. periods)	Collagen Yield (%)	Elemental % Composition (C)	Elemental % Composition (N)	C:N Atomic Mass Ratio
H6	12522	Tschetter Site Prairie Side No BP	17	317	62	30	11.15	44.1	15.4	3.3
H7	12523		17	323	59	26	8.44	42.7	14.6	3.4
H8	12524		18	335	65	26	8.51	42.7	15.6	3.2
H9	12525		17	338	68	30	21.42	30.5	11.6	3.1
H10	12526		17	352	57	40	13.77	45.6	16.1	3.3
H11	12527		16	302	58	22	8.90	43.6	15.4	3.3
I1			16	333	59	34	20.33	47.9	17.1	3.3
I2		Oklahoma, Modern	16	333	63	34	24.18	30.4	10.8	3.3
I3			15	355	77	34	27.91	50.9	16.2	3.7
J1		Saskatoon, Modern	15	328	59	34	21.91	49.7	18.0	3.2
J4			17	320	61	32	21.06			
J5		Yorkton, Modern	18	320	60	32	21.47			
J6			18	300	57	32	24.07			

*Values Are Aggregates

* Equipment Failure

Appendix F. REPORTED STABLE ISOTOPE RATIOS AND UNUSED SPECIMENS

F.1 Introduction

All measured stable isotope ratios for a given substance must be standardized and corrected. The accurate measurement of hydrogen stable isotope ratios also requires that a further calculation be made to correct for the impact of hydrogen exchange. Throughout the analysis, some samples may fail to meet the criteria for acceptance. Once the final values have been determined and these inappropriate samples have been removed from consideration, the final analysis of the reportable data may occur. The finalized stable carbon, nitrogen and hydrogen isotope ratios that were used during the analysis presented in this thesis are detailed in Table F1. All modern and prehistoric bison bone collagen samples that met the criteria for acceptance are listed (see Appendix E). Stable isotope ratios that were determined for several modern bison dietary samples are also shown at the end of the table. These values were not used in the subsequent analyses (see below).

In addition, this table also presents stable oxygen isotope ratios that were determined for all of the samples. Although these data are not formally analyzed in this project, they are presented here for posterity. Theoretically, stable oxygen isotope ratios should yield data relevant to the same issues that may be explored through the analysis of stable hydrogen isotope ratios. The main advantages of stable hydrogen isotope analysis for the examination of herbivore tissues, over that of stable oxygen isotope analysis, are that the stable isotopes of hydrogen exhibit larger mass differences than those of oxygen and that the hydrogen isotope composition of animal tissues derive from only two major environmental sources, diet and drinking water (see Chapter 1). Despite these important distinctions, the oxygen data may have presented an interesting comparison to that of the hydrogen. The main reason that these data were not analyzed was that little to no previous research regarding stable oxygen isotope compositions in

collagen have ever been undertaken. In addition to making any important comparisons impossible, this situation made it somewhat difficult to calibrate the laboratory equipment properly to insure accurate measurements.

Table F1. Summary Table of Reportable Stable Isotope Ratios.

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	$\delta^{13}C$ (PDB, ‰)	$\delta^{15}N$ (AIR, ‰)	δD (VSMOW, ‰)	$\delta^{18}O$ (VSMOW, ‰)	Comments
A2	12451	Heron Eden Site (EeOi-11), Cody Complex, 9150 BP	-19.8	6.1	-126	37.2	
A5	12454		-19.2	5.9	-128	36.4	
A6	12455		-19.8	5.5	-126	35.2	
A8	12457		-19.5	6.3	-131	36.0	
A9	12458		-19.2	6.5	-120	36.7	
A10	12459		-20.0	6.2	-132	35.2	
A11	12460		-20.1	5.8	-129	35.9	
C4	12471	Norby Site (FaNq - 56), Mummy Cave, 7050 BP	-19.5	7.2	-117	36.0	
C5	12472		-18.4	8.2	-124	36.0	
C7	12474		-19.2	7.7	-123	35.6	
C11	12478		-18.9	6.9	-123	35.1	
C12	12479		-19.4	8.4	-115	35.2	
C13	12480		-19.3	8.2	-115	35.4	
C14	12481		-18.4	8.7	-102	35.9	
B1	12462	Gowen Sites (FaNq - 32), MummyCave, 5850 BP	-19.2	7.1	-142	34.2	
B2	12463		-19.3	8.9	-126	34.9	
B3	12464		-18.4	8.3	-129	35.5	
B4	12465		-18.8	7.8	-139	37.1	
B5	12466		-19.5	9.8	-124	37.2	
B6	12467		-20.0	7.3	-129	35.6	
D1	12482	Amisk (FbNp - 17), Oxbow, 4350 BP	-19.9	8.2	-125	35.1	
D2	12483		-19.2	7.6	-120	34.2	
E1	12484	Harder Site (FbNs - 1), Oxbow, 4200 BP	-19.1	6.4	-117	35.6	
E4	12487		-19.1	7.7	-144	36.5	
E5	12488		-19.2	8.2	-140	36.6	
E6	12489		-16.5	6.4	-118	35.8	
E7	12490		-19.0	7.8	-125	36.8	
E8	12491		-19.2	7.5	-137	36.4	
E11	12494		-19.6	7.4	-136	34.4	
E12	12495		-18.8	7.3	-140	33.7	
F1	12499	Thundercloud Site (FbNp - 25), McKean, 3400 BP	-19.5	8.1	-146	35.4	
F2	12500		-18.5	7.2	-126	35.9	
F3	12501		-20.2	7.2	-129	36.4	
G1	12502	ald Site (ElNp - 8), sant, 1550 BP	-17.5	7.8	-107	37.9	
G2	12503		-18.5	7.4	-124	34.6	
G3	12504		-18.6	8.1	-107	35.1	
G7	12508		-19.1	8.5	-124	35.5	
G8	12509		-18.3	7.7	-124	35.7	
G10	12511		-18.0	7.8	-110	36.2	
G12	12513		-18.5	8.4	-114	36.7	

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	$\delta^{13}C$ (PDB, ‰)	$\delta^{15}N$ (AIR, ‰)	δD (VSMOW, ‰)	$\delta^{18}O$ (VSMOW, ‰)	Comments
G13	12514	Fitzger Be	-17.0	7.4	-108	35.2	
G14	12515		-16.2	7.6	-110	35.6	
G15	12516		-16.2	6.5	-125	37.2	
H1	12517	Tschetter Site (FbNr - 1), Prairie Side Notched, 1050 BP	-19.8	7.0	-137	34.7	
H2	12518		-19.7	5.2	-126	35.0	
H3	12519		-19.6	6.3	-119	35.8	
H4	12520		-17.7	6.5	-112	36.3	
H5	12521		-17.6	6.1	-110	35.5	
H6	12522		-19.4	7.5	-119	35.1	
H7	12523		-18.1	6.8	-123	36.7	
H8	12524		-19.3	7.0	-112	36.1	
H11	12527		-18.8	6.3	-128	35.9	
I1	*Values Are Aggregates	Oklahoma, Modern	-13.3	3.2	-62	39.2	
I2			-15.7	4.9	-59	39.0	
I3			-15.0	3.1	-80	38.3	
J1		Saskatoon, Modern	-21.6	5.2	-130	33.4	
J4			-23.0	5.6	-144	33.8	
J5		Yorkton, Modern	-22.9	6.8	-124	34.8	
J6			-22.2	4.1	-124	34.6	
Dietary Samples							
I4	*Values Are Aggregates	Oklahoma, Modern	-18.2	14.1	-103	44.6	Stomach Contents
J2		Saskatoon, Modern 2001	-26.5	2.5	-128	47.2	Feed
J3			-26.8	4.3	-184	43.8	Grass
J7		Saskatoon, Modern 2002	-29.1	2.6	-188	42.0	Grass
J8			-27.0	7.8	-156	47.7	Feed
J9		Yorkton, Modern 2002	-28.6	7.7	-215	42.3	Stomach Contents
J10			-27.2	2.1	-189	43.5	Grass

F.2 The Modern Bison Dietary Samples

Along with the modern bison bone collagen, diet samples associated with some of the modern bison were also collected. The stable isotope values that were determined for these samples are presented in Table F2 along with those of the tissues from the individual bison with which they are associated. While the modern bison bone collagen stable isotope ratios were analyzed during this study in an attempt to demonstrate the range of expected tissue δD values between two geographically distinct locations (see Chapter 2), the values from the dietary components were not evaluated. The diet samples were initially collected to provide insight into the relationship between the δD composition of bison diet and bison bone collagen. Unfortunately, this objective was felt to be beyond the scope of this thesis. These values are thus, presented here for posterity only.

Table F2. Associations Between Modern Bison Bone Collagen Samples and Dietary Samples.

Sample ID		Site, Cultural Association, Approximate Age (R.C.Y.)	$\delta^{15}N$ (‰)	$\delta^{13}C$ (‰)	δD (‰)	$\delta^{18}O$(‰)	Comments
I1		Oklahoma, Modern	3.2	-13.3	-59.2	39.2	Bone Collagen
I2			4.9	-15.7	-55.5	39.0	Bone Collagen
I3		Oklahoma, Modern	3.1	-15.0	-76.4	38.3	Bone Collagen
I4			14.1	-18.2	-102.1	44.6	Stomach Contents (* For Sample I3)
J1	2001	Saskatoon, Modern	5.2	-21.7	-135.4	33.4	Bone Collagen
J2			2.5	-26.5	-128.4	47.2	Feed (* For Sample J1)
J3			4.3	-26.8	-183.7	43.8	Grass (* For Sample J1)
J4	2002	Saskatoon, Modern	5.6	-23.0	-148.9	33.8	Bone Collagen
J7			2.6	-29.1	-188.5	42.0	Grass (* For Sample J4)
J8			7.8	-27.0	-156.4	47.7	Feed (* For Sample J4)
J5		Yorkton, Modern	6.8	-22.9	-129.3	34.8	Bone Collagen
J6			4.1	-22.2	-129.3	34.6	Bone Collagen
J9		Yorkton, Modern	7.7	-28.6	-216.4	42.3	Stomach Contents (* For Samples J5,J6)
J10			2.1	-27.2	-188.9	43.5	Grass (* For Samples J5, J6)

Appendix G. CALCULATED C_4 PERCENTAGES

G.1 Introduction

During the analysis presented in this thesis, the diets of prehistoric bison were inferred and compared using $\delta^{13}C$ values measured from bone collagen samples. Since $\delta^{13}C$ values reflect the relative inputs of C_3 and C_4 plant species in bison diet, and because these plant species form two large groups which may be distinguished on the basis of the stable-carbon isotope composition of their tissues, it is possible to calculate a rough estimate of the proportion of C_4 plants that an animal has consumed over the better part of its life. Table G1 records the estimated percentage of C_4 plants calculated from the $\delta^{13}C$ values measured in the bone collagen of individual bison examined in this thesis. These calculations were based upon the following equation (Schwarcz *et al.* 1985):

$$C_4\,\% = \frac{(\delta^{13}C_{\text{measured}} - \delta_3 - \Delta_{dc})}{\delta_4 - \delta_3} \times 100\%$$

where $\delta^{13}C_{\text{measured}}$ is the measured carbon isotope composition of an animal's bone collagen, δ_3 and δ_4 represent the average $\delta^{13}C$ values from within the range exhibited by most C_3 plants (-26.5‰) and most C_4 (-12.5‰) plants respectively, and Δ_{dc} represents the average trophic fractionation (5‰) between an animal's diet and its bone collagen (Tieszen 1991).

Table G1. Calculated C_4 Percentages For Reported Samples

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	$\delta^{13}C$ (PDB, ‰)	Calculated C_4 Percentage
A2	12451	Heron Eden Site (EeOi-11), Cody Complex, 9150 BP	-19.8	12.1
A5	12454		-19.2	16.4
A6	12455		-19.8	12.1
A8	12457		-19.5	14.3
A9	12458		-19.2	16.4
A10	12459		-20.0	10.7
A11	12460		-20.1	10.0
		Mean	-19.7	13.2
		Std. Dev.	0.4	2.6
C4	12471	Norby Site (FaNq - 8), Mummy Cave, 7050 BP	-19.5	14.3
C5	12472		-18.4	22.1
C7	12474		-19.2	16.4
C11	12478		-18.9	18.6
C12	12479		-19.4	15.0
C13	12480		-19.3	15.7
C14	12481		-18.4	22.1
		Mean	-19.0	17.8
		Std. Dev.	0.5	3.3
B1	12462	Gowen Sites (FaNq - 32), MummyCave, 5850 BP	-19.2	16.4
B2	12463		-19.3	15.7
B3	12464		-18.4	22.1
B4	12465		-18.8	19.3
B5	12466		-19.5	14.3
B6	12467		-20.0	10.7
		Mean	-19.2	16.4
		Std. Dev.	0.6	4.0
D1	12482	Amisk (FbNp - 17), Oxbow, 4350 BP	-19.9	11.4
D2	12483		-19.2	16.4
		Mean	-19.6	13.9
		Std. Dev.	0.5	3.5
E1	12484	Ns - 0 BP	-19.1	17.1
E4	12487		-19.1	17.1

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	$\delta^{13}C$ (PDB, ‰)	Calculated C_4 Percentage
E5	12488	Harder Site (Fb 1), Oxbow, 420	-19.2	16.4
E6	12489		-16.5	35.7
E7	12490		-19.0	17.9
E8	12491		-19.2	16.4
E11	12494		-19.6	13.6
E12	12495		-18.8	19.3
		Mean	-18.8	19.2
		Std. Dev.	1.0	6.9
F1	12499	Thundercloud Site	-19.5	14.3
F2	12500	(FbNp - 25),	-18.5	21.4
F3	12501	McKean, 3400 BP	-20.2	9.3
		Mean	-19.4	15.0
		Std. Dev.	0.9	6.1
G1	12502	Fitzgerald Site (ElNp - 8), Besant, 1550 BP	-17.5	28.6
G2	12503		-18.5	21.4
G3	12504		-18.6	20.7
G7	12508		-19.1	17.1
G8	12509		-18.3	22.9
G10	12511		-18.0	25.0
G12	12513		-18.5	21.4
G13	12514		-17.0	32.1
G14	12515		-16.2	37.9
G15	12516		-16.2	37.9
		Mean	-17.8	26.5
		Std. Dev.	1.0	7.3
H1	12517	Tschetter Site (FbNr - 1), Prairie Side Notched, 1050 BP	-19.8	12.1
H2	12518		-19.7	12.9
H3	12519		-19.6	13.6
H4	12520		-17.7	27.1
H5	12521		-17.6	27.9
H6	12522		-19.4	15.0
H7	12523		-18.1	24.3
H8	12524		-19.3	15.7
H11	12527		-18.8	19.3
		Mean	-18.9	18.7
		Std. Dev.	0.9	6.3

Sample ID	Lab ID	Site, Cultural Association, Approximate Age (R.C.Y.)	$\delta^{13}C$ (PDB, ‰)	Calculated C_4 Percentage
I1	*Values Are Aggregates	Oklahoma, Modern	-13.3	58.8
I2			-15.7	41.4
I3			-15.0	46.3
		Mean	-14.7	48.8
		Std. Dev.	1.3	8.9
J1	*Values Are Aggregates	Saskatoon, Modern	-21.6	0.0
J4			-23.0	0.0
J5		Yorkton, Modern	-22.9	0.0
J6			-22.2	0.0
		Mean	-22.4	0.0
		Std. Dev.	0.7	0.0