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Stable isotope evidence for dietary patterns and environmental conditions at Tell Leilan, Syria, ca. 1900-2900 B.C.

by

Rebecca Susanne Godkin Feasby

A thesis submitted to the Faculty of Graduate Studies and Research
in partial fulfillment of the requirements for the degree of Master of Arts

Department of Anthropology

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FACULTY OF GRADUATE STUDIES AND RESEARCH

The undersigned certify that they have read, and recommend to the Faculty of Graduate Studies and Research for acceptance, a thesis entitled *Stable isotope evidence for dietary patterns and environmental conditions at Tell Leilan, Syria, ca. 2900-1900 B.C.* submitted by *Rebecca Susanne Godkin Feasby* in partial fulfillment of the requirements for the degree of *Master of Arts*.

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January 26, 1998

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ABSTRACT

Stable carbon and nitrogen isotope ratios in preserved protein from prehistoric bone have been used extensively to trace the dietary patterns of ancient human groups. The isotopic analysis of 16 individuals, ranging in age from birth to adult, from the site of Tell Leilan, Syria, revealed a terrestrial-based diet largely composed of C_3 plants, with the additional consumption of meat and legumes. Methodological investigations suggested that a weak acid solution provides the best means of isolating protein from poorly preserved skeletal samples.

Some researchers have suggested that at approximately 2200 B.C., a marked drying trend induced considerable degradation of land-use conditions at and around the site of Tell Leilan, which apparently led to an occupational hiatus that lasted for roughly 300 years. Chronological comparisons of isotope ratios in this study, however, do not indicate that the diet of these people changed between 2900 and 1900 B.C.

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Chapter 1

Introduction

For the past two decades, a group of archaeologists has been working in a remote region of Syria trying to piece together the cultural history of a lost civilization. In doing so, these scientists have stumbled upon a rather mysterious research problem that has far-reaching consequences. After bringing together archaeological data, historical records of population movement, and analyses of soil moisture from the site in Syria, Harvey Weiss and his colleagues were convinced that the society of Tell Leilan (Figure 1.1) abruptly fell apart at the end of the third millennium B.C. due to a drying trend that swept across northern Mesopotamia. They have argued that, after four centuries of productive urban life, the city of Tell Leilan collapsed leaving vestiges of a shattered society (see Weiss, 1996; Weiss et al., 1993). Evidence of concurrent collapse in adjacent regions suggests that the impact of the presumed climatic change was extensive (Weiss, 1996; Weiss and Courty, 1993) and has added another dimension to this controversial archaeological question.

In order to determine the exact nature of the abandonment at Tell Leilan, it is necessary to understand the subsistence patterns and environmental conditions that supported the population before its collapse. The objective of this study is to analyze human skeletal remains from Tell Leilan for the stable isotopes of carbon and nitrogen and to use this information to reconstruct the general dietary adaptations and environmental conditions at Tell Leilan at and before the time of its collapse.

By tracing the patterns of food consumption in prehistoric human groups, researchers are able to extend our capacity for understanding ancient economic systems, cultural practices, and disease profiles. The reconstruction of ancient dietary patterns from bone chemistry has been an integral part of anthropological inquiry since the late 1970s (Price et al., 1985. See DeNiro and Epstein, 1978; Schoeninger, 1979; van der Merwe and Vogel, 1978; Vogel and van der Merwe, 1977). In particular, the isotopic analysis of carbon and nitrogen has provided researchers with a relatively accurate means of obtaining dietary information from prehistoric bone. Early laboratory and field studies affirmed that the isotopic composition of an animal's diet is recorded in the isotopic structure of the animal's tissues (DeNiro and Epstein, 1978; DeNiro and Epstein, 1981). The levels of carbon and nitrogen in bone collagen are under strict metabolic control and the ratio of the stable isotopes of each of these elements ($^{13}C/^{12}C$ and $^{15}N/^{14}N$) provides a direct window onto the dietary habits of individuals (Price et al., 1985).

Most elements exist in nature as combinations of two or more isotopes that have the same number of nuclear protons but different numbers of neutrons. Although the different isotopes of a particular element have the same general chemical properties, the rates of chemical reaction differ among stable isotopes due to their different masses. The isotopically heavier forms of a particular element move slower than the lighter isotopes during chemical reactions and these different rates of movement are preserved chemically in organic tissues such as bone. In anthropological research, the mass-dependent isotopic

signatures of carbon and nitrogen are used to distinguish dietary patterns in humans and animals.

The differences in rates of movement between specific isotopes result in discrimination or fractionation, such that isotopic forms are typically incorporated differentially into the products of a chemical reaction. Isotopic fractionations that occur during chemical reactions involving carbon and nitrogen are generally very small, such that the differences are expressed in fractions of a percent (parts per thousand or ‰). The measurement scheme used to determine stable isotope abundances involves calculating the ratio of the heavier to the lighter isotope, with reference to the ratio of a laboratory standard. Isotope ratios are thus expressed in delta (δ) units:

$$\delta(‰) = [(R_{sample}/R_{standard}) - 1] \times 1000.$$

where R is the ratio of the heavier to the lighter isotope (Ambrose, 1993; Katzenberg, 1992). Since the carbon isotope standard (a marine limestone fossil) contains more of the heavier isotope (^{13}C) than nearly all dietary resources, $\delta^{13}C$ values are typically negative. Conversely, $\delta^{15}N$ are generally positive because most food resources contain more of the heavier isotope (^{15}N) than the nitrogen isotope standard (atmospheric air).

Researchers have generally agreed that collagen is the preferred material for dietary analysis using stable isotopes since it comprises approximately 20% of bone by weight and is remarkably resistant to post-mortem degradation (see Ambrose, 1993, 1990; Chisholm et al., 1983). Sample preparation for isotopic analysis involves demineralizing the bone in dilute hydrochloric acid (HCl) or ethylenediamine-tetraacetic acid (EDTA) and then freeze-drying the organic residue (collagen) (see Amrose, 1993 and Katzenberg, 1992 for reviews). An additional step is often included involving treating the demineralized bone with sodium hydroxide (NaOH) to remove organic contaminants such as humic acids (e.g. Ambrose, 1990; Moore et al., 1989; Sealy et al., 1995). The collagen samples are then combusted to form CO_2 and N_2. The gases are introduced into a mass spectrometer where the ratios of the stable isotopes of carbon and nitrogen are recorded relative those of the laboratory standards.

Chemical and physical changes that occur in the burial environment can alter the biological information that is preserved in organic tissues. Microbial activity (see Grupe et al., 1989) and other diagenetic forces can result in biased $\delta^{13}C$ and $\delta^{15}N$ ratios in bone collagen. Postmortem loss of collagen and chemical degradation of the chemical properties of collagen can both be detected using elementary laboratory procedures. Collagen yields can be calculated by comparing the dry bone weight with the weight of the freeze-dried collagen sample. Scientists typically reject collagen samples that are less than 1% of the original dry bone weight due to the possibility of isotopic enrichment (see Ambrose, 1990; Schoeninger et al., 1989; Schwarcz and Schoeninger, 1991). The purity of bone collagen samples can also be evaluated by determining the carbon to nitrogen ratios for each sample. Based on laboratory examinations of animal specimens with known feeding habits, DeNiro (1985) proposed that bone collagen samples with C/N ratios between 2.9 and 3.6 are suitable for the identification of feeding habits. Collagen samples with C/N ratios that fall outside of this range are suspect due to the possibility of contamination or the presence of non-collagenous proteins.

In cases where the integrity of the collagen sample is questionable, the determination of the amino acid composition of the collagen extract can be used to verify the chemical nature of the demineralized bone. The amino acid profile for collagen is unique due to the relatively large amounts of glycine and proline and the presence of hydroxyproline and hydroxylysine (Katzenberg, 1992; see also Katzenberg, 1989 for an application). By determining the amino acid composition of the collagen residue, the quality of the sample can be ascertained.

The stable isotope ratios measured by mass spectrometric analysis can be used to interpret dietary adaptations in humans and animals and can provide researchers with a means of discerning palaeoenvironmental conditions. Dietary reconstructions using stable isotopes are possible because certain food items have unique isotopic ratios that are preserved in human and animal tissue. C_4 (e.g. tropical grasses such as maize and millet) and C_3 plants (e.g. temperate grasses such as wheat and barley; fruits and nuts) each have a distinct isotopic composition with average $\delta^{13}C$ values of -12.5‰ and -26.5‰ respectively (Ambrose, 1993). Thus, it is possible to determine the dietary source of carbon using stable isotope ratios based on this C_3/C_4 dichotomy. Stable carbon isotopes have been used to trace prehistoric changes in diet (e.g. Little and Schoeninger, 1995; Schwarcz et al., 1985; van der Merwe and Vogel, 1978; White and Schwarcz, 1989), to determine social status based on preferential access to certain foods (e.g. Ubelaker et al., 1995), and to determine the relationship between diet and disease (White and Armelagos, 1997). $\delta^{13}C$ values can also be used to distinguish between marine and terrestrial foods based on the 7‰ difference between marine and terrestrial carbon sources (Chisholm et al., 1982; During, 1997; Lidén, 1995; Lovell et al., 1986; Mays, 1997; McGovern-Wilson and Quinn, 1996; Pate, 1997; Sealy and van der Merwe, 1988, Tauber, 1981).

Stable nitrogen isotopes have also proven to be useful indicators of dietary adaptations. In particular, $\delta^{15}N$ values have been used to distinguish marine from terrestrial dietary sources (e.g. Larsen et al., 1992), to determine weaning age in infants (e.g. Fogel et al., 1989; Katzenberg, 1993; Katzenberg et al., 1996), and to decipher trophic level differences within food chains (e.g. Tuross et al., 1994).

Researchers have also suggested that it may be possible to use stable nitrogen isotopes to assess the level of water-stress in humans and animals and to, therefore, ascertain specific climatic conditions such as rainfall levels (Ambrose, 1986). In a survey of nitrogen isotope ecology in South Africa, Sealy et al. (1987) found that animal $^{15}N/^{14}N$ ratios varied with rainfall, with higher $\delta^{15}N$ values in more arid regions. Ambrose and DeNiro (1989) analyzed herbivore tooth collagen from Holocene Africa and found that $\delta^{13}C$ and $\delta^{15}N$ variations throughout this period were directly linked to environmental conditions such as rainfall. Similarly, Lee-Thorp and Beaumont (1995) used $^{13}C/^{12}C$ ratios to determine seasonal shifts in rainfall during the late Quaternary in South Africa. Environmental reconstructions may also be possible based on the different growing conditions required by C_3 and C_4 plants: C_3 plants prefer a moist environment, whereas C_4 plants are well adapted to dry growing conditions.

The use of stable carbon and nitrogen isotopes in estimating prehistoric environmental and dietary conditions has the potential to be particularly useful in

evaluating the effects of climatic change. In agricultural regions, the land-use effects of an abrupt climatic change can result in significant alterations in soil moisture reserves, wind velocities, precipitation patterns, and ground visibility. These changes, in turn, can coincide with conditions of low agricultural productivity or crop-switching. For a society dependent on agricultural resources, an abrupt climatic change could translate into critical losses of valuable land and could ultimately lead to the displacement of a population.

Several researchers have argued that at the end of the third millennium B.C., many regions of the ancient Near East experienced such an abrupt climatic change. According to these interpretations, synchronous climatic changes in Mesopotamia, the Aegean, Egypt, Palestine, and the Indus (Weiss, 1996; Weiss et al., 1993) led to the eventual collapse of states dependent on agriculture. Recent excavations at the ancient site of Tell Leilan in Syria have convinced several researchers that a desertification trend at around 2200 B.C. induced a considerable degradation in land-use conditions and led to the abandonment of Tell Leilan and the collapse of surrounding socio-political entities and the Akkadian Empire.

Archaeological investigations at the site of Tell Leilan have revealed a large third millennium B.C. city that, unlike its southern counterparts, relied on rain-fed agriculture (Weiss, 1986). Without a network of irrigation canals to support agricultural production during dry spells, northern Mesopotamian centres such as Tell Leilan would have been crippled by drought. Soil moisture studies and stratigraphic analyses have shown that at approximately 2200 B.C., subsequent to a volcanic eruption, northern Mesopotamian civilization came to an abrupt end (Weiss, 1996, 1994; Weiss et al., 1993). Historical records suggest that, in search of food and water, the northern settlers moved south. Unable to support the huge influx of people, the southern Mesopotamian centres experienced sudden and severe urban chaos (Gibbons, 1993) and, as a result, collapsed. Weiss and his colleagues (1993; see also Weiss and Courty, 1993) have suggested that the disintegration of northern Mesopotamian cities and the subsequent collapse of the Akkadian empire can be directly linked with an abrupt climatic change that resulted in desertification.

Weiss' ideas have come under considerable attack in the past several years (e.g. Rosen, 1997; Yoffee, 1995). Researchers have criticized his attempt to link climatic change with the fall of an empire as being overly broad and generalized, and as lacking the necessary quantitative data to confirm his hypothesis. Several researchers at Harvard and at the Centre Nationale de Recherche Scientifique in Paris are engaged in ongoing palaeoethnobotanical studies, faunal analyses, and geochemical studies aimed at reconstructing the palaeoenvironment of Tell Leilan at the time of its collapse. Under the guidance of Weiss at Yale, these scientists are attempting to generate the fundamental quantitative data required to reconstruct the environmental conditions that led to the collapse and abandonment of the northern Mesopotamian civilization. The isotopic analysis of skeletal remains from the site of Tell Leilan may provide additional support for Weiss' climatic change hypothesis and may confirm his notion that the third millennium northern Mesopotamian civilizations literally dried up and withered away. If rains became scarce and winds swept across the nothern Mesopotamian plains during the

mid third millennium B.C., isotopic analysis should reveal elevated $\delta^{13}C$ and $\delta^{15}N$ values during a period of drought.

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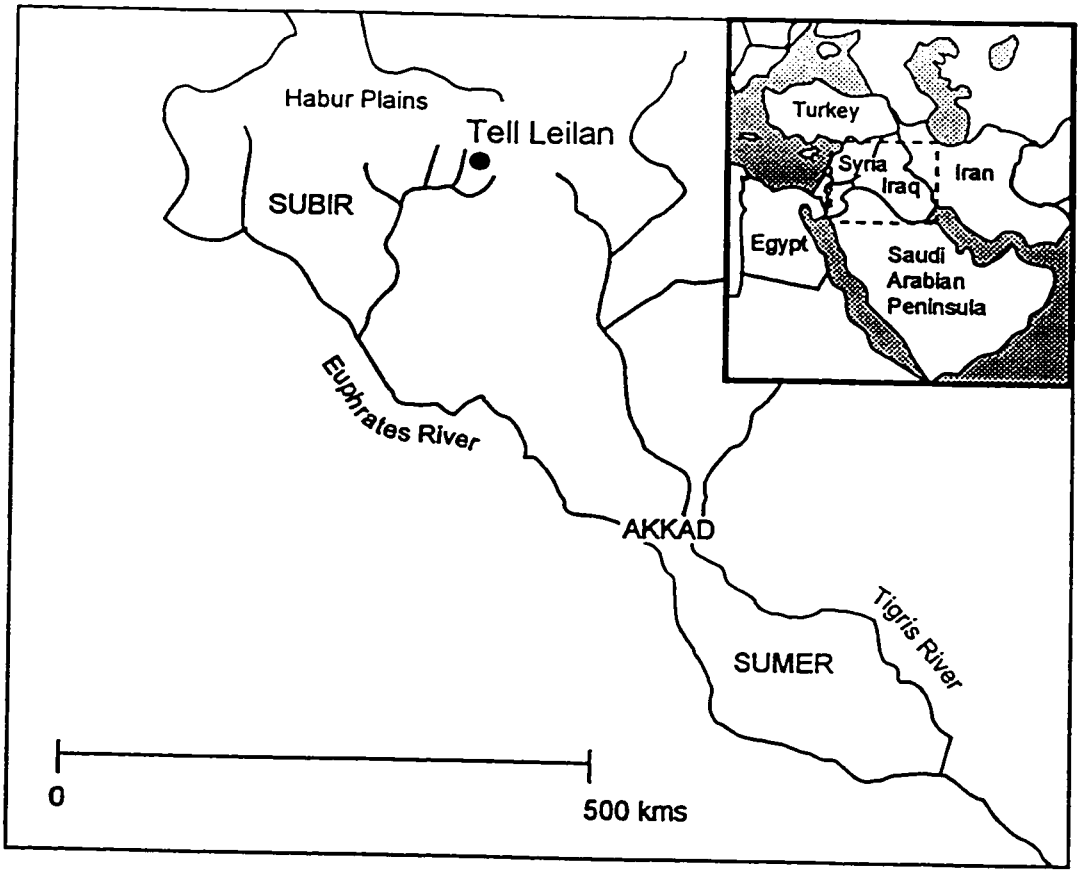
Figure 1.1. Map of ancient Mesopotamia showing the location of Tell Leilan (adapted from Gibbons, 1993)



Chapter 2

The origin and collapse of the Akkadian Empire and the archaeological significance of Tell Leilan

Introduction

More than five millennia ago, the world's first complex societies arose in the fertile alluvial plains of the Near East, in what is now Iraq. These civilizations evolved in a physical environment that demanded increased organization and resource management to overcome severe climatic difficulties. In antiquity, the fertile plains and valleys drained by the Euphrates and the Tigris offered the richest potential farmland between the Indus and the Nile. During the mid-fourth millennium B.C., the early empires of Mesopotamia emerged in this dry, undulating region, which challenged its inhabitants to control an unpredictable water supply and manage agricultural production. In southern and central Mesopotamia, the construction of elaborate dams and canals enabled farmers to create an irrigation system that could support large-scale agriculture and the growth of urban centres. During the fourth millennium, cities in this region grew as large as 100 hectares and, by the middle of the third millennium B.C., urbanization had reached new heights (Adams, 1981; Weiss, 1986a). The enormous populations of these early cities supported the agricultural work force required to tend the large network of irrigation systems necessary to sustain continued urban development, enhance trade, and maintain large armies. This urban flourescence in southern Mesopotamia appeared in the context of the exceptional productivity of irrigation agriculture, which was a consequence of soil conditions, irrigation technology, rainfall. and the social organization of labour as ration-paid workers (Weiss, 1986a).

While the development of southern Mesopotamian civilization and agriculture has been studied in-depth for the past several decades (e.g. Adams, 1972; Oppenheim, 1964), the origin and growth of cities in the dry-farming northern reaches of Mesopotamia has only recently received significant attention (Weiss, 1986a). This incognizance is mostly due to the lack of early archaeological excavations in this area. Accordingly, the political and economic history of ancient northern Mesopotamia was not written until the mid 1980s (Weiss, 1985b). As research efforts heightened and the archaeological and epigraphical documentation improved, the historical significance of northern Mesopotamian cities became clearer. Archaeological investigations on the Habur Plains, in particular, have contributed to the development of a chronological and developmental framework specific to the urban centres of northern Mesopotamia (Weiss, 1990c).

During the early stages of Mesopotamian urbanization, southern rulers took control of massive tracts of land that spanned from the Persian Gulf to the headwaters of the Euphrates in modern-day Turkey (Gibbons, 1993). At the centre of this stronghold was Sargon of Akkad, who instituted the Akkadian policies of expansion and conquest (Oppenheim, 1964). Cuneiform texts have revealed that the Akkadians secured long distance trade networks and established imperial rule over the northern centres (Drower

and Bottéro, 1971). In northern Mesopotamia, pre-existing cities were expanded and fortified and agricultural production was intensified. As archaeological excavations unfolded in the northern sites, researchers discovered that, unlike the southern Mesopotamians, the northern settlers did not rely on irrigation works to control agricultural production. Instead, the northern Mesopotamians developed an efficient dry-farming system that relied on rainfall and did not require a high input of labour. Although rain-fed agriculture provided significant labour-saving advantages, farming in northern Mesopotamia was far less predictable than irrigation agriculture in southern Mesopotamia. Environmental disturbances, such as drought, could have spelled serious trouble in the dry-farming regions of northern Mesopotamia. The site of Tell Leilan in northeastern Syria (Figure 2.1) has provided much of the information for researchers attempting to understand the mechanisms of early urban expansion in northern Mesopotamia and has supplied archaeologists with a wealth of information on the potential effects of climatic change in the ancient Near East.

In this review, the cultural and archaeological history of Mesopotamia and Tell Leilan will be presented and the anthropological significance of the site of Tell Leilan will be examined. The consequence of climatic change in these early civilizations will be contemplated and the novel research problem being investigated at Tell Leilan will be discussed. Accordingly, this review will survey the importance of archaeological research in northern Mesopotamia and consider the environmental significance of the collapse of early civilizations, such as that at Tell Leilan.

A brief cultural history of Mesopotamia and Tell Leilan

The rise of the world's first complex societies was centred on the ancient floodplains of the Euphrates, in southern Mesopotamia. The origin and early history of the third millennium B.C. cities and states of Sumer and Akkad have been a major focus of archaeological research for the past century (Weiss et al., 1993). As in any period or locality, the phenomenon of urbanization in Mesopotamia depended on the institution of the state as a political form and was constrained by various social and ecological processes, such as specialization of labour, control of production, and technological innovations (Adams, 1972). However, perhaps the most important determinant of early urban patterns of development was geography: the land, in essence, shaped the city.

The pattern of agricultural land use in ancient Mesopotamia was controlled by the river regimes and climate. In southern Mesopotamia, the distribution pattern of river water necessitated the control of agricultural crops and rain water by irrigation canals. The southern Mesopotamians accomplished this by constructing fan-like irrigation channels that allowed for the cultivation of large tracts of soil considerably inland from the Tigris and Euphrates (Oates and Oates, 1976). The establishment of irrigation-based farming practices that controlled such massive stretches of land demanded a large labour force. Grand cities, ruled by kings, developed as centres of agricultural and political control (Weiss, 1985c). Archaeologists have successfully traced the developmental history of these southern territories, including the evolution of state-level political

organization. In doing so, researchers have attempted to sketch the pattern of secondary state formation in this region and to analyze the nature of the political and economic control based in southern Mesopotamia.

The middle of the third millennium B.C. marks the apogee of southern Mesopotamian urbanization (Adams, 1981; Weiss, 1986b; see Table 2.1 for Mesopotamian chronology). During this period, populations peaked and an urban hierarchy became firmly established. From approximately 2300 to 2200 B.C., southern Mesopotamia was united under the imperial rule of Sargon of Akkad and his dynastic successors (Weiss et al., 1993). Under Akkadian reign, irrigation-based agricultural production in southern Mesopotamia was imperialized and southern power was expanded to include the rain-fed agricultural regions to the north. The growth of Akkad's northern neighbour, Subir, affirms the cultural and political influence that the southern administrative centres extended to the northern Mesopotamian states (Weiss, 1990a; Weiss et al., 1993). Weiss (1990a) has suggested that a commodity interest, such as mineral resources, may have attracted southern Mesopotamian attention to the Habur Plains of Subir, while others have indicated that urbanization in Mesopotamia was merely gathering momentum (e.g. Adams, 1981). Indeed, both agricultural production and imperial state formation were intensifying throughout Mesopotamia and, thus, as a long-term agricultural system, the large irrigation-based cities seemed to be sowing "the seeds of their own destruction" (Weiss, 1986b, p. 71; see also Jacobsen, 1982).

The overwhelming concentration of people in large cities at the mid-third millennium in Mesopotamia has been regarded by some researchers as "a hypertrophic, 'unnatural' condition for an agricultural civilization with preindustrial transport technology" (Adams, 1981, p. 138). By controlling agricultural production and trade in the north and surrounding areas, however, the southern Mesopotamians could have secured the surplus from the relatively high-yield dry-farming on the Habur Plains to feed the growing population. Southern governors administered agricultural operations in the northern centres and fortresses were built to control imperial wheat production (Weiss, 1996). The Akkadian Empire also maintained firm trade control of commodities from Anatolia, Lebanon, and the Gulf of Oman (Weiss, 1996). Imperial taxes provided a rich source of income for the southern rulers and the Akkadians prospered like never before.

Stratigraphic analyses at the site of Tell Leilan in northern Mesopotamia have indicated that a major transformation of settlement occurred on the Habur Plains at the middle of the third millennium B.C. (Weiss, 1985a), at around the same time as urban development peaked in the south. At approximately 2600-2500 B.C., three major urban centres developed equidistant from each other in Subir. The equidistance between these centres has raised fundamental questions for understanding the structure of third millennium rain-fed agriculture state systems: were these urban centres optimizing the availability of stream locations, rainfall, soils, and topography?; or was each of these cities exerting territorial control over the immediately surrounding area? (Weiss, 1992).

Before the emergence of these large urban centres, the rain-fed plains of northern Mesopotamia were dotted with dispersed agricultural communities (Weiss, 1983a) that utilized the fertile dry-farming environment of the Habur Plains for barley and wheat

cultivation (Weiss, 1983b). Following the mid-third millennium expansion of Mesopotamia, settlement patterns on the Habur Plains were radically altered with the sudden emergence of state-level society, as seen in the large planned city of Tell Leilan (Weiss et al., 1993). Excavations have revealed that simple village economies were replaced by structured economies based on central collection and the storage-and-redistribution of agricultural products and animal goods (Weiss et al., 1993). The sudden development of cities such as Tell Leilan at around 2600 B.C. suggests that the agricultural centres of the Habur Plains underwent autonomous regional urban growth prior to the establishment of the Akkadian dynasty (Senior and Weiss, 1992) rather than being imposed militarily by southern Mesopotamia (Weiss, 1990b). Archaeological remains suggest that this urban growth continued unfettered for approximately 300 years, until the northern centres of the Habur Plains were incorporated into the Akkadian imperial structure at approximately 2300 B.C.[1] (Weiss, 1990b; Senior and Weiss, 1992).

The growing northern urban centres such as Tell Leilan eventually became the focus of southern Mesopotamian attack (Drower and Bottéro, 1971; Weiss, 1990c). Sargon of Akkad set a pattern of royal behaviour that centred on the conquest of strategically important regions and the institution of imperial rule over neighbouring areas (Oppenheim, 1964). Together with his large armies, Sargon of Akkad stormed the northern Mesopotamian plains and established a new imperial bureaucracy to control wheat production and monitor the collection of taxes (Weiss, 1996). The northern cities were fortified to protect imperial land claims and, at Tell Leilan, a city wall was erected and 'palace' storage rooms were constructed (Weiss, 1990b). River courses were diverted to support the increase in agricultural production generated by imperial control (Weiss, 1994) and ration-paid workers were deployed across the northern Mesopotamian landscape (Weiss, 1985c). Under Akkadian rule, the northern centres continued to grow and southern Mesopotamia benefited from imperial taxes and agricultural surplus. In terms of prosperity and abundance, urban life had reached new heights in the Near East. At approximately 2200 B.C., however, misfortune befell the peoples of Mesopotamia.

Archaeological excavations at Tell Leilan have revealed a period of inactivity between 2200 and 1900 B.C. This occupational hiatus has been defined by the collapsed remains of Akkadian buildings covered with erosion deposits at each sounding at the site of Tell Leilan (Weiss, 1991; Weiss, 1996; Weiss et al., 1993). Towards the end of the third millennium B.C., urban activity in the Akkadian controlled northern centres apparently came to an abrupt end, leaving remnants of shattered cities across the Habur Plains and throughout northern Mesopotamia (Gibbons, 1993; Weiss and Courty, 1993). According to historical records, agricultural production ceased and the northern

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[1] The installation of Akkadian political and economic control in the northern urban centres of the Habur Plains remains an active area of investigation. The nature of the productive relations between the southern administrative centres and the northern dry-farming municipalities is largely ill-defined and poorly understood. However, the political relation between northern and southern Mesopotamia will likely become more apparent as research at Tell Leilan and surrounding areas continues.

Mesopotamians abandoned their settlements and headed south. Scribes in the royal courts in Akkadia described on clay tablets an influx of northern "barbarians" and the construction of a wall to hold them back (Gibbons, 1993). The new arrivals from the north strained food and water supplies and wreaked havoc on the already densely populated southern urban centres. The collapse of the Akkadian Empire, it seems, started in the north with the abrupt abandonment of cities across the Habur Plains (see Weiss et al., 1993), and ended in the south with urban chaos.

The collapse of these ancient Mesopotamian cities has puzzled researchers and has resulted in the proposal of various hypotheses to explain the sudden abandonment of urban settlements in northern Mesopotamia. The archaeological team working at the site of Tell Leilan has offered an explanation that has resulted in a considerable amount of controversy. According to Harvey Weiss and his colleagues, the Akkadian Empire's fall was triggered by an abrupt climatic change that resulted in desertification of the northern Mesopotamian plains and surrounding regions:

> Sometime around 2200 B.C. seasonal rains became scarce, and withering storms replaced them. The winds cut through northern wheat fields and blanketed them in dust. . . .For more than a hundred years the desertification continued, disrupting societies from southwestern Europe to central Asia. (Weiss, 1996, p. 31).

In the dry-farming regions around Tell Leilan, drought would have meant disaster. The Akkadian imperial ventures, which were sustained in the pre-existing northern agricultural centres, would have fallen apart and the southern Mesopotamians would have lost one of their principal sources of wealth.

However, many archaeologists and epigraphers have been reluctant to blame Akkad's collapse on sudden climatic change. Norman Yoffee has suggested, instead, that internal dissension and political strife were responsible for the fall of Akkad:

> . . .stretching the resources of the city-states resulted in massive resistance on the part of the local rulers and traditional elite, who, in the end, were able to resist the demands of the central administration and to bring it down (Yoffee, 1995, p. 293).

Joseph Tainter (1988) has asserted that single-event catastrophes, such as volcanic eruptions and sudden climatic change, are "too simple to accommodate the complexities of human societies and the collapse process" and that "it is doubtful if any large society has ever succumbed to a single-event catastrophe" (p. 53). Again, refuting the notion of sudden collapse, Adams (1988) has implicated political struggle in the collapse of Akkad, suggesting that north-south regional differences became exacerbated into almost national differences within Mesopotamia, resulting in the dissolution of city-states. Other researchers have explained the collapse of northern Mesopotamian cities in terms of "hyperurbanization" in the regions around Tell Leilan (e.g. Wilkinson, 1994).

Weiss and other proponents of the climatic change hypothesis have prompted a reappraisal of archaeological data and have cited new geoclimatic figures in support of an abrupt climatic change in ancient Mesopotamia. Microscopic examinations of soils dating from 2200 to 1900 B.C. showed a great deal of windblown dust, little evidence of earthworm activity, and other signs of aridity that suggested reduced rainfall and soil erosion (Weiss, 1996; Weiss et al., 1993). Weiss and his colleagues have also noticed an astonishing trend of collapse during the late third millennium B.C. in civilizations ranging from Crete and mainland Greece, to Harappa and Mohenjo-daro in the Indus Valley (Weiss, 1996).

The lack of irrigation works uncovered in northern Mesopotamia, combined with historical documentation, points to a region dependent on rain-fed agriculture – one that would have been crippled by a sudden change in climate that resulted in desertification. According to Weiss (1996), flight would have been a good option for the peoples of the Habur Plains: by abandoning urban life and migrating to areas where agriculture was still sustainable – the irrigation-based cities of Akkad and Sumer – the inhabitants of northern Mesopotamia would have stood a chance at survival.

Archaeology at Tell Leilan

While working for the British Museum in the late 1800s, Hormuzd Rassam supervised a series of archaeological investigations in northern Mesopotamia in search of spectacular ancient sites and valuable antiquities (Weiss, 1981). In 1878, while surveying in what is now northeastern Syria, Rassam spotted the site of Tell Leilan rising above the horizon. Rassam, however, passed by Tell Leilan and focused instead on excavations in southern Mesopotamia. It was not until nearly one hundred years later that the site of Tell Leilan became a focus of archaeological inquiry.

The occupational history of Tell Leilan was first investigated in 1978 by a team of archaeologists from Yale University led by Harvey Weiss (see Weiss, 1983a). During the first field season, archaeologists began a systematic survey of "a site which seems certain to alter our understanding of ancient Near Eastern history" (Weiss, 1981, p.22). By 1980, Weiss and his colleagues had ascertained that Tell Leilan was occupied at least as early as the fifth millennium B.C., had been a large town of no more than 15 hectares until about 2500 B.C., and had then suddenly expanded to a staggering 90 hectares encompassed by an enormous city wall (Weiss, 1984). These early excavations were among the first to prove that urban expansion at Leilan and across the Habur Plains of northern Mesopotamia was nearly synchronous with similar developments already documented for the southern Mesopotamian alluvium (e.g. Adams, 1981).

Following the initial archaeological surveys of Tell Leilan, Weiss and his colleagues turned their attention to more specific problems, including the origins and collapse of cities and states across the Habur Plains towards the end of the third millennium B.C. and the probable identification of Tell Leilan with ancient Šubat Enlil, one capital of the "Great Kingdom of Upper Mesopotamia" under the reign of Šamši-Adad (1813 - 1781 B.C.) (Weiss et al., 1990; see Kupper, 1973 for a review of this time

period). Archaeological findings during the 1987 excavation season confirmed the presence of an 18th century B.C. elaborate palace and administrative materials suggested that the palace was likely erected during the reign of Šamši-Adad (Akkermans and Weiss, 1989). The 1989 and 1991 seasons of field research were designed to retrieve additional data for the classification of Tell Leilan as the second millennium B.C. site of Šubat-Enlil and for understanding mid-third millennium urbanization and state formation (Weiss, 1990b, 1994).

The 1991 field season witnessed the completion of the first stage of the Leilan research project. Beginning in 1993, the focus of archaeological investigations at Tell Leilan shifted toward an examination of landscape and climatic data for Leilan and the surrounding region during "Habur hiatus I", ca. 2200-1900 B.C., when the Habur Plains area of northern Mesopotamia was apparently abandoned (Weiss, 1994). Archaeological surveys have failed to identify any ceramic assemblages for this period and surface reconnaissance studies of Tell Leilan and neighbouring cities have indicated a widespread occupational hiatus beginning at approximately 2200 B.C. (Weiss et al., 1993). Together with a colleague from Paris, Weiss began analyzing soil samples in order to determine the basis of this gap in the archaeological record. Analyses of soil layers showed that a volcanic eruption at around 2200 B.C. may have ushered in a period of drier, less hospitable conditions.[2] Microscopic examinations of soil samples revealed tell-tale signs of aridity – fewer earthworm holes and wind-blown pellets and dust (Gibbons, 1993; Weiss et al., 1993). Studies of ocean sediments between Greenland and Iceland have revealed a cold peak at around 2200 B.C. that, according to one climatologist, would have caused a dry spell in southern Europe and western Asia (see Weiss, 1996). Additionally, lake cores taken from Western Tibet have indicated a long-term trend toward aridity that culminated around four thousand years ago (Gasse et al., 1996). These researchers have suggested that the environmental fluctuations recorded in Tibet are "in phase with climatic changes recognized in tropical North Africa" and have proposed that the dry events at the end of the third millennium B.C. were caused by "abrupt disequilibrium in the climatic system" (Gasse et al., 1996, p. 79). In an ongoing, and as yet inconclusive, research project, American geoclimatologists are examining cores from the floor of the Gulf of Oman in search of dust sediments that would suggest an abrupt climate change in the Near East towards the end of the third millennium B.C. (see Weiss, 1996).

Global and local climates change and shift in a broad spectrum of cycles and time ranges (Mörner, 1987). The climate of desert-belt regions appears to be particularly susceptible to change and the climate and soil regimes of these regions are typically regarded as fragile (Otterman and Starr, 1995). The climate history of the Middle East is

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[2] Major volcanic eruptions have been considered to influence global weather patterns for some time (Pyle, 1992), although researchers have generally been reluctant to attribute regional climatic change solely to volcanic activity (Weiss, 1996). Instead, researchers have ususally opted for more inclusive theories of climatic change that incorporate geologic models, palynological sampling, and sea level analyses (COHMAP, 1988).

known to have varied considerably during the past 10,000 years and researchers have often attributed this variability to the region's intermediate position between the humid Mediterranean and the arid desert belt (Issar, 1995; see Nasrallah and Balling, 1995 for a review of recent changes).

Nevertheless, many Near Eastern archaeologists have been reluctant to blame the fall of civilizations solely on climatic change and have, instead, explained societal collapse in terms of political struggle, agricultural overintensification, and increased social complexity (e.g. Adams, 1988; Rosen, 1995; Tainter, 1988; Tainter, 1995; Wilkinson, 1994). According to several researchers, systemic, short-term climatic variability may have intensified societal instability at times of overpopulation, but an abrupt and severe climate change could not solely account for the collapse of a civilization (see Wilkinson, 1994). Other researchers have countered Weiss' ideas by maintaining that societal collapse in the ancient Near East was not necessarily an unavoidable response to climatic change, since later agricultural states apparently functioned within the same drier environmental setting (e.g. Rosen, 1995, 1997).

Catastrophe scenarios, such as *abrupt* climatic change, have historically been favoured as explanations for societal collapse (Tainter, 1988). However, these sort of explanations have recently come under increased attack for oversimplifying the process of collapse and for underestimating the ability of complex societies to withstand catastrophes (e.g. Adams, 1988; Tainter, 1988; Tainter, 1995). Proponents of abrupt-climatic-change hypotheses, such as Weiss, contend that the Mesopotamian geologic and archaeological evidence paint a rather sensational picture of a civilization that succumbed to the pressures of a harsh and abrupt change in climate. According to Weiss, an abrupt climatic change was the misfortune that brought Akkad to its knees.

By adopting a "catastrophic" approach to uncovering the archaeological significance of the collapse of Mesopotamia, Weiss and his colleagues have developed a theoretical framework that underscores the significance of chance events in human history. In the case of Tell Leilan, the storms of the third millennium represent an historical catastrophe that had devastating and far-reaching effects. Weiss and other champions of the abrupt-climatic-change theory maintain that "drought, wind, and dust" – not restless political rulers or military conquest – resulted in the destruction of one of the world's first complex civilizations (Weiss, 1996, p. 30).

Conclusion

The site of Tell Leilan is shrouded in mystery. Until 2200 B.C., the archaeological record contains remnants of temples, towns, and administrative buildings. The third millennium growth of these urban centres was, in many instances, unparalleled and populations exploded everywhere. Massive city walls were erected as southern imperial rule extended its reach over northern Mesopotamian settlements and administrative texts reveal extensive trade networks and the organization of agricultural production. Before long, this prosperity apparently drew to an abrupt close. The archaeological record of Tell Leilan is bare between 2200 and 1900 B.C. The collapsed

buildings of the third millennium city are covered with erosion deposits that show no trace of human activity for approximately 300 years (Weiss, 1996). For Weiss (1997), late third millennium collapse in northern Mesopotamia was a social adaptation to an unmanageable set of environmental conditions.

The bizarre occupational hiatus at Tell Leilan and the nature of the collapse of the Akkadian Empire remains subject to debate and the climatic change hypothesis generated by archaeological research at Tell Leilan has disrupted the complacent consensus that has surrounded traditional interpretations of civilizational collapse (Weiss, 1996; see also Adams, 1981 and Tainter, 1988). As soil analyses and palaeoclimatological studies continue, data on the climate of northern Mesopotamia at the end of the third millennium B.C. will illuminate the situation at Tell Leilan at the time of its collapse.

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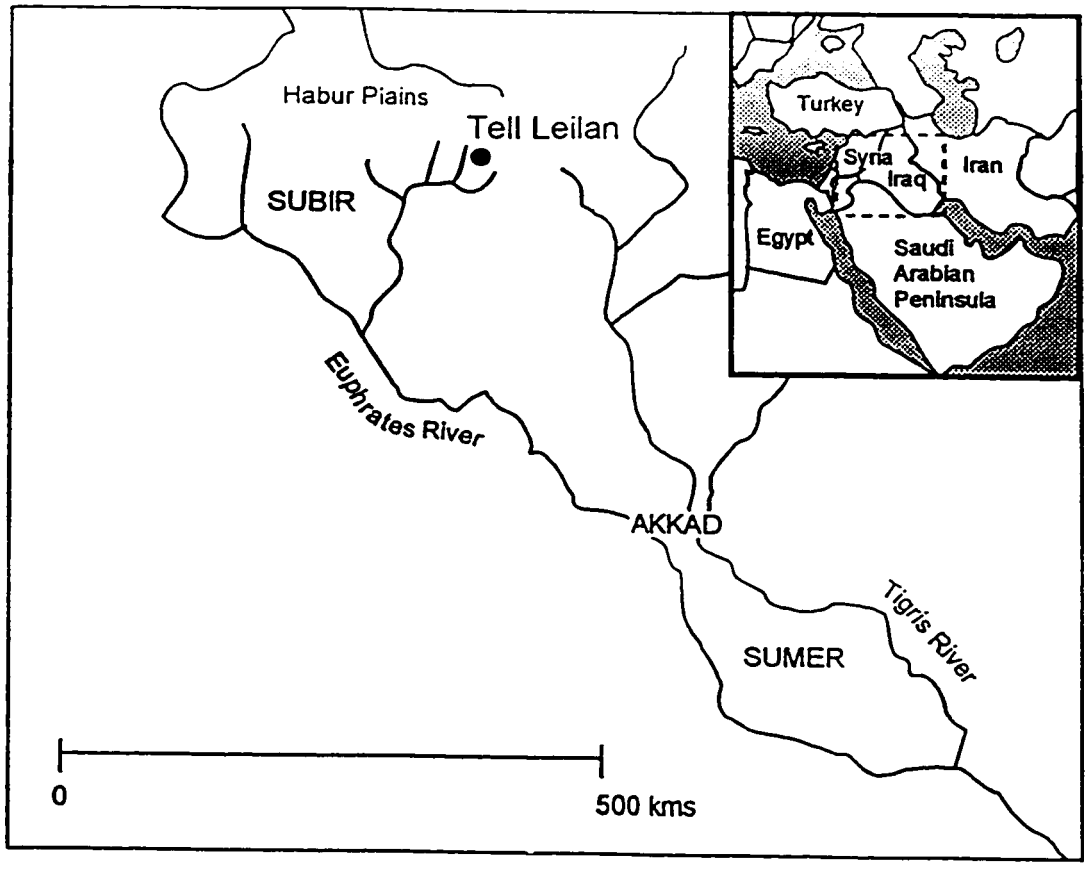
Table 2.1. Third millennium Mesopotamian chronology (modified from Weiss et al., 1993)

ca. B.C.	Tell Leilan (Habur Plains)	Southern Mesopotamia	Developmental Stage
1900	I	Old Babylonian	Post-abandonment reoccupation of Mesopotamia
	....	....	
2000	Habur hiatus	Isin-Larsa	Desertification of northern Mesopotamia and abandonment throughout Mesopotamia
2100	I	Ur III	
2200	....	Guti	
	IIb	....	Akkadian conquest of northern Mesopotamia; imperialization
2300	....	Akkad	
		....	
2400	IIa	ED IIIb	Consolidation of state power in northern Mesopotamia
2500	IIId	ED IIIa	
2600		Late ED II	Secondary state formation on the Habur Plains; massive urban expansion
2700	IIIc	Early ED II	
2800	IIIb	ED I	Structured cities in southern Mesopotamia; smaller villages and towns throughout northern Mesopotamia
2900	IIIa	Jemet Nasr	
3000	IV	Late Uruk	

Figure 2.1. Map of ancient Mesopotamia showing the site of Tell Leilan on the Habur Plains (adapted from Gibbons, 1993)



Chapter 3

Understanding prehistoric human diet using stable carbon and nitrogen isotope ratios of bone collagen

Introduction

The study of variation and change in human diets is important in several respects. First, it allows anthropologists to consider relationships between diet and patterns of population size, density, distribution, growth, and mobility and to consider dietary trends in cultural evolution. Second, it paves the way for a more complete understanding of the environmental conditions that support particular dietary adaptations. Finally, when applied to prehistoric societies, the study of human diets provides significant insight into ancient subsistence strategies and can provide clues about environmental conditions, social divisions of labour, settlement and trading patterns, and nutritional diseases. Stable isotope analysis of prehistoric bone collagen offers one quantitative method of diet reconstruction which, when combined with other analytical techniques, extends our capacity for investigating the lives of ancient peoples.

The use of osteochemical techniques to evaluate prehistoric food consumption is a relatively recent innovation in physical anthropology and archaeology. Early analyses of the geochemistry of stable carbon isotopes (Craig, 1953) and the relationship between carbon isotope ratios and photosynthesis (Bender, 1971) provided much of the impetus for consequent isotopic studies of palaeodiets. This research documented the differential discrimination of the heavier isotope of carbon by C_3 and C_4 plants during photosynthesis. Since humans ultimately derive their carbon from the plants that they consume, it seemed reasonable to expect that the isotopic values of dietary plants might be preserved in human tissues. In essence, researchers began testing the assumption that you are, in fact, what you eat.

The potential for reconstructing prehistoric human diets using stable isotope analysis was fully realized in the late 1970s with the innovative work of Vogel and van der Merwe (1977), van der Merwe and Vogel (1978), and DeNiro and Epstein (1978). Using stable carbon isotope ratios of bone collagen from human skeletal material in New York State, Vogel and van der Merwe (1977) demonstrated a method for detecting the consumption of maize based on different carbon isotopic ratios between pre-horticultural hunter-gatherers and horticulturists. Van der Merwe and Vogel (1978) later quantified long term consumption of maize among Woodland humans from eastern North America and demonstrated that maize was an insignificant dietary component in this area prior to AD 1000. At the same time, DeNiro and Epstein (1978) investigated the relationship between diet and carbon isotope ratios in animal tissues using controlled feeding experiments. Their subsequent examination of nitrogen isotopes (DeNiro and Epstein, 1981) confirmed their initial findings with carbon isotopes and provided categorical support for the notion that the isotopic signatures of dietary resources are reflected in the isotopic composition of bone collagen.

The early promise of stable isotopes as reliable indicators of prehistoric consumption patterns generated a tremendous increase in the range of applications and resulted in a proliferation of studies aimed at refining the methodology and expanding the practical scope of isotopic analysis (reviewed by Ambrose, 1993; Chisholm, 1989; Katzenberg, 1992a; Kazenberg and Harrison, 1997; Keegan, 1989; Schoeninger and Moore, 1992; Schwarcz and Schoeninger, 1991). Stable isotope applications have broadened to include the estimation of the dietary importance of marine versus terrestrial food resources (Chisholm et al., 1982; During, 1997; Larsen et al., 1992; Lovell et al., 1986b; Mays, 1997; McGovern-Wilson and Quinn, 1996; Pate, 1997; Schoeninger and DeNiro, 1984; Schoeninger et al., 1983; Sealy and van der Merwe, 1988; Tauber 1981; Walker and DeNiro, 1986), ecological reconstructions (Ambrose and DeNiro, 1987; Ambrose and DeNiro, 1989; Gröcke, 1997; Sealy et al., 1987; van der Merwe, 1989), the determination of class differences (Iacumin et al., 1996; Ubelaker et al., 1995), the estimation of human dependence on animal versus plant protein (Ambrose, 1986; Ambrose and DeNiro, 1986; Schwarcz et al., 1985), the determination of weaning age (Fogel et al., 1989; Katzenberg, 1993; Katzenberg and Pfeiffer, 1995; Katzenberg et al., 1993; Katzenberg et al., 1996; Schurr, 1997; White and Schwarcz, 1994;), and the detection of disease in ancient populations (White and Armelagos, 1997).

This review will present the basic chemical principles that underlie stable isotope analysis and discuss briefly the technology behind isotope ratio mass spectrometry. The use of bone collagen as the preferred material for isotopic analysis will be examined, as well as problems typically encountered in the reconstruction of diet from stable isotope ratios, including diagenesis and analytical difficulties. The methods developed to isolate bone collagen will be briefly reviewed, paying particular attention to the usefulness of certain techniques. The fractionation of carbon and nitrogen in nature will be reviewed in short, providing a framework for subsequent discussions of stable isotope applications in archaeology. Finally, the methodology employed in this study will be described.

Chemical theory, nomenclature, and presentation of stable isotope values

Isotopes are varieties of chemical elements that have the same number of protons, but different numbers of neutrons. Since the numbers of protons and neutrons in an element account for its atomic mass, the mass number for an isotope of a particular element varies according to the number of neutrons in its nucleus. The extra weight in 'heavier' isotopes results in slower chemical reaction rates relative to the 'lighter' isotopic forms. These differences in rates of movement and reaction result in discrimination or *fractionation*, such that the isotopic forms are typically incorporated differentially in the products of a chemical reaction. *Stable* isotopes are, by definition, not radioactive and do not change in abundance over time. Archaeologically important elements, such as carbon and nitrogen, occur naturally as two different isotopically stable varieties: ^{12}C and ^{13}C for carbon and ^{14}N and ^{15}N for nitrogen. Together, carbon and nitrogen constitute the building blocks of organic molecules (Keegan, 1989) and the

mass-dependent differences in carbon and nitrogen abundance in organic human tissues has provided the foundation for palaeodietary studies.

The measurement scheme for stable isotopes in palaeodietary studies involves calculation of the ratio of the heavier to the lighter isotope, with reference to the ratio of a standard laboratory material. Isotope ratios are expressed using the delta (δ) notation in fractions of a percent (parts per thousand, or per mil., = ‰):

$$\delta X(‰) = [(R_{sample}/R_{standard}) - 1] \times 1000$$

where δX is the ratio of the two sample isotopes relative to the standard and R is the ratio of the heavier to the lighter isotope (Ambrose, 1993; Katzenberg, 1992a). For carbon, the standard is a marine carbonate fossil (*Belemnitella americana*) from the Peedee formation in South Carolina and is referred to as PDB. The standard reference material for nitrogen is atmospheric air (AIR). Because the carbon isotope standard contains more ^{13}C than nearly all dietary resources and human tissues, $\delta^{13}C$ values are typically negative. Conversely, $\delta^{15}N$ values are usually positive since the nitrogen isotope standard has less ^{15}N than most food resources.

Mass spectrometry

A mass spectrometer measures the atomic mass of an element (Masterton and Hurley, 1989). By measuring the number of ions of a given mass, the spectrometer can measure the relative abundance of each isotope of a particular element (Kotz and Purcell, 1991). Mass spectrometric analyses in anthropology and archaeology typically follow the following general protocol: approximately 5 mg of freeze-dried bone collagen is placed in quartz tubes with cupric oxide, elemental copper, and silver foil, following the procedure of Stump and Fraser (1973), as modified by Sofer (1980), and described by Chisholm (1989) and Ambrose (1990). The tubes are vacuum-sealed and heated to combust the samples to form CO_2 and N_2. The gases are then directed into the mass spectrometer alternately with the standard reference gases. The gases are ionized and then accelerated through a mass analyzer (usually a magnet) that differentially deflects heavy and light ions. The light ions are deflected more than heavier ones and by comparing the accelerated voltages required to bring the two ions to the same point, it is possible to determine the relative masses of the ions and, accordingly, the relative abundances of ions of different masses (Masterton and Hurley, 1989). Once the procedure is complete, a computer prints out the ratio of stable isotopes in both the sample and standard gases and then calculates a δ value, which can be used for palaeodietary reconstructions.

Human bone collagen

Structure and amino acid composition of collagen

As one of the most stable and plentiful organic components of vertebrate hard tissues, collagen is necessarily of primary concern to those interested in fossil bones and

teeth. Collagen accounts for approximately 90 to 95 percent of the total protein content in bone (Eyre, 1980; Price et al., 1985; Tuross et al., 1980) and is the preferred organic material for isotopic analysis because it constitutes approximately 20 percent of dry bone by weight (Ambrose, 1993). The majority of archaeological research on prehistoric bones has used collagen for isotopic analysis since this protein has been detected in fossil material dating as far back as the Jurassic Period (Wyckoff, 1980). At least five genetically distinct types of vertebrate collagen have been identified (Eyre, 1980). The collagen found in bone, skin, tendon, dentin, and fascia is referred to as Type I (Schwarcz and Schoeninger, 1991) and will be the focus of the following description.

Bone is a composite material made of protein and mineral in the form of hydroxyapatite (for reviews of the chemical composition of bone see Katzenberg, 1989, 1992a; Krueger and Sullivan, 1984; Schwarcz and Schoeninger, 1991; White, 1991). The mineral component gives bone its hardness and rigidity and, when soaked in acid to dissolve these minerals, bone becomes rubber-like and flexible. Extensive linkages between each of three equal-sized polypeptide chains (often called α chains) in the form of a triple helix give collagen its great tensile strength and make it relatively insoluble. Each α chain is composed of approximately 1000 amino acid residues and is about 300 nanometres in length (Eyre, 1980). Approximately two-thirds of these residues are formed from only four amino acids: glycine (333/1000 residues) , alanine (110/1000 residues), proline (131/1000 residues), and hydroxyproline (89/1000 residues). The presence of hydroxyproline and another amino acid, hydroxylysine, serve to distinguish collagen from other proteins and the high frequency of glycine and the presence of hydroxyproline and hydroxylysine are used as diagnostic features of collagen.

Diagenesis

Researchers have found that bone collagen is frequently preserved long after burial (DeNiro, 1985; Stafford et al., 1988) and that the loss of other proteins and trace elements from buried bone appears to be more complete than the loss of collagen (Hedges and Wallace, 1980; Nelson et al., 1986). Nevertheless, scientists often face the dilemma of assessing the biochemical integrity of collagen samples. Various post-mortem processes and conditions can threaten the purity of bone samples and may alter the isotopic signatures preserved in the organic fraction of bone. Accordingly, an understanding of post-depositional changes in bone structure and chemistry is necessary for precise and replicable results.

In bone chemistry, the concept of diagenesis refers to the chemical and physical alteration of bone following sedimentary deposition (Katzenberg, 1992a). Diagenetic alteration is assessed by anthropologists in order to verify the integrity of archaeological bone and to determine the extent to which the chemical and physical conditions of the burial environment may have modified bone collagen. Researchers have increasingly come to recognize the palaeodietary significance of diagenesis and the complex post-mortem history of fossil bone (see Schwarcz et al, 1989). In many instances, archaeological bone has been subjected to numerous post-burial processes that may result

in the gain or loss of chemical constituents, including the addition of such contaminants as humic acids and the loss of organic material (Sandford, 1992; Schwarcz and Schoeninger, 1991). Archaeological conservation and consolidation techniques may also affect the chemical properties of skeletal material through the addition of preservatives.

While the problem of diagenesis is rather serious for trace element analysis (Lambert et al., 1989; Lambert et al., 1990; Price et al., 1992; Sillen, 1981; see Sandford, 1992 for a review of diagenetic concerns in trace element analysis), diagenetic alteration appears to be less of a problem for stable isotope studies using bone collagen (Katzenberg, 1992a). Nelson and colleagues (1986) found that while stable isotope and strontium concentrations in the inorganic phase of bone were modified diagenetically and therefore not reliable dietary markers, the carbon and nitrogen composition of bone collagen was not altered by post-burial change. However, the authors cautioned against applying their findings to other archaeological contexts, submitting that the cool pre-excavation conditions of their archaeological samples may have contributed, in part, to the stability of the collagen analyzed. Indeed, other authors have suggested that cool, stable burial environments can, in large part, contribute to the viability of archaeological bone collagen (e.g. Ambrose, 1993).

In an examination of marine and terrestrial mammal bones, DeNiro (1985) tested the assumption that bone collagen isotope ratios are not affected by post-mortem processes. His results indicated that post-burial alterations of collagen isotope ratios do occur, but that prehistoric bones with carbon to nitrogen (C/N) ratios between 2.9 and 3.6 provide reliable samples for isotopic analysis and dietary reconstruction. Schoeninger and DeNiro (1982) provided additional criteria for determining the state of collagen preservation. They noted that isotopic ratios no longer reflect dietary isotopic proportions when the organic fraction of bone is experimentally reduced to less than 5 percent of its original dry weight (see also Schoeninger et al., 1989). This is a fairly generous standard given that historic human bones have collagen concentrations of 10.9 to 27.0 percent by weight (Ambrose, 1990).

In collagen samples contaminated with humic substances (base-soluble, organic products of plant decomposition), Katzenberg (1989) found higher C/N ratios than expected and $\delta^{13}C$ values that reflected the decaying plant matter present at the time of burial. Soaking demineralized bone in sodium hydroxide has proven a practical means of removing these humic acid contaminants (DeNiro and Epstein, 1981; Katzenberg, 1989; Katzenberg et al., 1995; Moore et al., 1989), although this method has also been found to remove a small fraction of collagen (DeNiro, 1985; Katzenberg, 1989; see Ambrose, 1990 and Boutton et al., 1984 for reviews).

Collagen extraction techniques

Since the first application of stable isotope analysis to prehistoric diet reconstruction (Vogel and van der Merwe, 1977), bone chemists have striven to clarify the logic and methodology of stable isotope studies. Accordingly, many attempts have been made to refine sample preparation methods. The isolation of collagen from fossil

bones has, in particular, received a great deal of attention. The three most common methods for the preparation of collagen are outlined below (see Moore et al., 1989 and Schwarcz and Schoeninger, 1991 for reviews).

Method 1

Developed by DeNiro and Epstein (1978, 1981), modified by Schoeninger and DeNiro (1984), and based on earlier work by Longin (1971), this method works well on fresh bone and well preserved archaeological material (Schwarcz and Schoeninger, 1991). Clean, dry bone is ground to a powder and 50 to 150 mg of crushed bone is demineralized in 1*M* HCl for 20 minutes and then filtered. The residue is soaked in 0.125*M* NaOH at room temperature for 20 hours, filtered, and rinsed with distilled water to neutrality. The residue is then gelatinized by soaking in 0.001*M* HCl and heating to 95°C for 10 hours. The collagen solution is filtered, evaporated, and lyophilized (freeze-dried) and the residual collagen is combusted to produce CO_2 and N_2 and analyzed with a mass spectrometer.

Method 2

This method was developed by Tuross and colleagues (1988) as an alternative to the first method for treating poorly preserved samples. In this procedure, 0.5 to 1.0 g of clean, dry bone is soaked in 0.5*M* EDTA (ethylenediamine-tetraacetic acid) buffered to pH 7.2 at 4°C for up to 5 days, during which time the sample is centrifuged and decanted and the EDTA solution is replaced daily (Moore et al., 1989). At the end of this period, the EDTA solution is poured off and the sample may be soaked in 0.125*M* NaOH for 20 hours, following which the sample is centrifuged and rinsed with distilled water to neutral pH. The remaining "organic residue" (Schwarcz and Schoeninger, 1991) is freeze-dried and then combusted to form CO_2 and N_2 and analyzed with a mass spectrometer.

Method 3

The third method, developed by Sealy (1986; see also Sealy and van der Merwe, 1986), is the simplest of the three methods discussed in this chapter. Cleaned fragments of bone (1 to 5 g) are placed in closed vials and soaked in dilute hydrochloric acid (1% HCl) at room temperature until the bone is translucent and flexible. The time required for complete demineralization varies according to sample size and the type of bone, and can range anywhere from several days to more than a month (Sealy, 1986). When demineralization is complete, the HCl is drained and the sample is rinsed with distilled water to neutral pH. The demineralized bone may then soaked in 0.125*M* NaOH for 20 hours and rinsed with distilled water to neutrality. The remaining insoluble 'pseudomorph' is freeze-dried and then combusted to produce CO_2 and N_2 and analyzed with a mass spectrometer.

The extreme ease and minimal laboratory requirements of the third method make it the obvious choice for many physical anthropology students and researchers with poor facilities and time constraints. However, several authors have pointed out one potential problem with this technique. Schoeninger and colleagues (1989) and Schwarcz and Schoeninger (1991) have warned that it can be difficult to determine when demineralization is complete. One solution to this problem is to dry and weigh the bone sample; the sample should be replaced in acid if the dry weight exceeds 25% of the original dry bone weight (Schwarcz and Schoeninger, 1991).

The objective in each of these procedures remains the isolation of collagen from the inorganic portion of bone and the elimination of organic contaminants. Archaeological bone samples selected for collagen extraction are typically rib fragments, which are least diagnostic for anatomical and pathological study (Schwarcz, 1991). There is some evidence to suggest that the 'pseudomorph' collagen extracted using whole bone chunks in HCl yields an amino acid profile more similar to that of a collagen standard (bovine collagen) than the bone powder method proposed by DeNiro and Epstein (1978, 1981) (Schoeninger et al., 1989). Additionally, Schoeninger and colleagues (1989) demonstrated that bone collagen obtained using method three contained nearly 100% protein, whereas the 'gelatin' collagen prepared from bone powder contained less than 10% protein. From this, they concluded that more than 90% of the 'collagen' extracted from bone powder was non-collagenous, indicating that the method for extracting collagen using dilute HCl and small chunks of bone is more likely to produce samples with "biological integrity" (Schoeninger et al., 1989).

Carbon and nitrogen fractionation in nature

Plants do not assimilate the isotopes of carbon equally, but discriminate against the heavier ones. It is known that plants belonging to the C_3 photosynthetic group, including wheat, rice, oats, root crops, legumes, vegetables, and most fruits, discriminate to a greater extent against ^{13}C than do C_4 plants. C_4 plants, such as maize, sugarcane, millets, and sorghum, evolved primarily in the tropics and are especially well adapted to high light intensities, high temperatures, and dry growing conditions. C_3 plants, with their less efficient and slower photosynthetic rate, prefer more shaded, cooler, and moister climates. The average $\delta^{13}C$ value for C_3 plants is about -27‰ and that for C_4 plants about -11‰ (Raven et al., 1986). A third photosynthetic pathway, known as Crassulacean Acid Metabolism (CAM), has evolved independently in many succulent plants, including cacti and pineapple. CAM plants are best adapted to very arid conditions where they are dependent on nighttime accumulations of carbon for their photosynthesis. Carbon isotope variation in CAM plants is a function of environmental conditions: in hot, arid environments C_4-like $\delta^{13}C$ values occur, whereas is cooler environments CAM plants have more negative, C_3-like $\delta^{13}C$ values (Ambrose, 1993).

One of many minerals essential for plant growth, nitrogen is taken up by plants from the soil and is obtained, by some plants, directly from the atmosphere. The isotopic ratio of soil nitrogen is greater than that of atmospheric nitrogen, resulting in lower $\delta^{15}N$

values for plants which fix atmospheric nitrogen (legumes) (Katzenberg, 1992a). Another category of plants, marine N_2-fixing blue-green algae, exhibit an enrichment in ^{15}N relative to terrestrial plants. This ^{15}N enrichment is carried up the food chain causing marine phytoplankton and fish to have greater $\delta^{15}N$ values than those of terrestrial plants and animals (Price et al., 1985; Schoeninger and DeNiro, 1984). Shifts in $\delta^{15}N$ values are evident at higher trophic levels as well. Herbivores show an increase of approximately +3‰ from the plants they consume, with legume-consuming herbivores having lower $\delta^{15}N$ values than those subsisting on a diet of nonleguminous plants (Katzenberg, 1992a); carnivores show an additional trophic level shift of about +3‰ (Schoeninger and DeNiro, 1984) (see Figure 3.1 for a summary of the distribution of $\delta^{13}C$ and $\delta^{15}N$ values in nature).

Using $\delta^{13}C$ and $\delta^{15}N$ values to solve prehistoric problems

Before reviewing several archaeological applications of stable isotope analyses, it is first necessary to examine how isotope ratios are affected by human metabolism. The use of stable carbon and nitrogen isotope ratios as palaeodietary indicators is founded on the assumption that the isotopic composition of bone collagen is a function of diet. However, in the course of metabolism, the isotopic ratios of food resources may be altered before the food energy is stored in human tissues (Ambrose and Norr, 1993; Keegan, 1989; van der Merwe, 1982).

In controlled feeding experiments with laboratory animals, DeNiro and Epstein (1978, 1981) found that both $\delta^{13}C$ and $\delta^{15}N$ values of whole bodies of animals were slightly more positive than those of their diets. Vogel and van der Merwe (1977) noticed a similar enrichment in $\delta^{13}C$ values from bone collagen and they estimated the magnitude of this effect to be approximately 5.1‰. Thus, for example, they calculated that the relative ^{13}C content of bone collagen in individuals subsisting entirely on C_4 plants would be approximately -7‰[1]. In a later study, van der Merwe and Vogel (1978) analyzed 31 archaic skeletons from several regions in woodland North America – almost exclusively a C_3 plant environment – and obtained an average $\delta^{13}C$ value of -21.4‰ ± 0.78‰. The thorough archaeological record of the diet of these peoples, which consisted of C_3 plants, terrestrial animals, and freshwater fish, and the consistency of their stable isotope measurements prompted the authors to calculate a fractionation factor for human bone collagen. Using a $\delta^{13}C$ value for C_3 plants of -26.5‰, van der Merwe and Vogel calculated a collagen enrichment factor of +5.1‰ (see also van der Merwe, 1982). The fractionation values typically accepted now are approximately +5‰ for $\delta^{13}C$ and about 2.5‰ to 3.0‰ for $\delta^{15}N$ (Keegan, 1989; Schoeninger, 1985, 1989).

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[3] This value was determined by taking the average $\delta^{13}C$ value of C_4 plants to be approximately -13‰ (range = -9 to -16) and adding a bone collagen enrichment factor of +6‰. They calculated a similar value for individuals subsisting solely on C_3 plants: -26‰ (average $\delta^{13}C$ value for C_3 plants – range from -22 to -34) + 6‰ (bone collagen enrichment factor) = -20‰.

In an extended examination of isotope fractionation during the production of collagen, Lovell and co-workers (1986a) examined the extent to which $\delta^{13}C$ values were affected by age- or sex-dependent physiological variables. Their results suggested that diet-to-collagen isotopic fractionation in human populations is not correlated with individual differences in age or sex. Moreover, their data indicated that the diet-to-collagen fractionation factor varies by no more than 0.3‰ and that any variation larger than this value must be attributable to dietary variability among individuals within the population (Lovell et al., 1986a).

Using carbon isotopes to distinguish dietary C_3 and C_4 plants

Initial applications of stable carbon isotopes in human prehistory developed as a response to early lines of research that suggested that the behaviour of carbon isotopes during photosynthesis could provide a tool for reconstructing the diet of ancient peoples (see Bender, 1968 and Calvin and Benson, 1948; see also van der Merwe, 1982). Most research using isotopic measurements to characterize diets based on C_3 or C_4 plants has focused on the timing of the introduction of maize, an important C_4 cultigen, in specific geographical regions.

The first isotopic evidence for early maize cultivation was interpreted by Vogel and van der Merwe (1977) in their examination of early horticulturists in New York State. They found that the $\delta^{13}C$ values for pre-horticultural hunter-gatherers were similar to those of Europeans, ranging from -21.3‰ to -19.8‰. The relative ^{13}C content of bone collagen from these individuals indicated a subsistence pattern that relied heavily on C_3 plants. The $\delta^{13}C$ values for horticulturists exhibited a significant isotopic change, with $\delta^{13}C$ values ranging from -13.5‰ to -16.6‰. The relative ^{13}C content of bone collagen from these individuals is consistent with a dietary adaptation that included C_4 plants; in this case, maize.

Using similar methods, Schwarcz and colleagues (1985) traced the consumption of maize in ancient native populations from southern Ontario. They noted a marked increase in the consumption of C_4 plants between AD 400 and 1100, with a shift in $\delta^{13}C$ values from -21.1‰ to -15.3‰ (see also Katzenberg, 1992b and Katzenberg et al., 1995). In their analysis of ancient Maya diet, White and Schwarcz (1989) found that maize persisted as a major dietary staple from pre-Classic to Historic times, but decreased in importance at one point during this period. Based on stable carbon isotope analyses of bone collagen from Late Woodland burials in New England, Little and Schoeninger (1995) suggested an early arrival of maize along the coast, but did not confirm that maize was a significant dietary component. Although $\delta^{13}C$ values (-9.6‰ to -11.0‰) were compatible with a diet consisting largely of C_4 plants, the lack of archaeological evidence supporting widespread cultivation indicated that maize was probably grown in small gardens or traded (Little and Schoeninger, 1995). Ubelaker and colleagues (1995) found a correlation between intensification of maize production and the development of a social hierarchy among individuals from precontact highland Equador. The $\delta^{13}C$ values

indicated that high-status individuals (mean = -10.3‰) were consuming more maize than low-status individuals (mean = -11.6‰).

Discriminating between diets based on marine and terrestrial resources

A further application of stable carbon isotopes to palaeodietary reconstruction is founded on the discrepancy in $\delta^{13}C$ values between marine and terrestrial resources. The basis of this application is the 7‰ difference between seawater biocarbonate and atmospheric CO_2 (Craig, 1953): dissolved bicarbonate (HCO_3) in seawater has a $\delta^{13}C$ value of approximately 0‰, whereas the main source of carbon for terrestrial plants and animals, atmospheric CO_2, has a $\delta^{13}C$ value of -7‰ (Katzenberg,1992a). This difference is expressed in the $\delta^{13}C$ values obtained for marine- and terrestrial-based diets, which are typically -14.5‰ to -16‰ for marine diets and -19‰ or less for terrestrial diets (Katzenberg, 1992a).

Tauber (1981) was the first to apply this observed fractionation in a study aimed at distinguishing the marine and terrestrial dietary habits of Mesolithic and Neolithic populations in Denmark. The results indicated that, whereas Mesolithic peoples relied heavily on marine resources, Neolithic populations subsisted primarily on terrestrial foods. In a later study, Lovell and colleagues (1986b) traced the prehistoric consumption of salmon by peoples who lived along rivers of interior British Columbia. Their $\delta^{13}C$ values indicated a trend for decreasing salmon consumption with increasing distance upstream. Sealy and van der Merwe (1988) also demonstrated the utility of stable carbon isotope ratios for tracing the role of marine foods over several thousand years and their results revealed changes in the pattern of marine food use during the Holocene in the south-western Cape of South Africa. Other studies aimed at characterizing prehistoric consumption of marine and terrestrial resources include those by Chisholm et al. (1982) on the proportions of marine- and terrestrial-based protein in aboriginal diets on the British Columbia coast; by Schoeninger and DeNiro (1984) on the carbon isotope composition of bone collagen from marine and terrestrial animals; by Walker and DeNiro (1986) on prehistoric dietary dependence on marine and terrestrial resources in California; by McGovern-Wilson and Quinn (1996) on the proportion of marine resources in the Afetna diet; by Mays (1997) on the relative contribution of marine resources to the diet of Mediaeval peoples of northern England; and by Pate (1997) on the contribution of marine and terrestrial foods to the diet of aboriginal Australians.

Stable nitrogen isotope ratios may also be used to discriminate between diets based on marine and terrestrial plant resources. Marine plants have $\delta^{15}N$ values that are approximately 4‰ higher than terrestrial plants, such that less positive $\delta^{15}N$ values are often indicative of a terrestrially based diet. Schoeninger and colleagues (1983) noted that prehistoric Eskimo and Northwest Coast Indian groups dependent on marine food sources showed higher $\delta^{15}N$ values than ancient agricultural groups from the same region. In an examination of ancient dietary change in the Georgia Bight, Larsen and colleagues (1992) found a decreasing emphasis on marine resources over time based on a temporal trend for less positive nitrogen isotope ratios.

Climate and habitat reconstruction

Carbon and nitrogen isotope ratios may also be used to determine palaeoclimatic and palaeoenvironmental conditions. Heaton and colleagues (1986) demonstrated a climatic influence on $\delta^{15}N$ values for human and animal bones from South Africa and suggested that stable nitrogen isotope ratios may be a useful tool for studying past climatic variations, particularly in regions where there have been large changes in precipitation. In a response to this report, Ambrose and DeNiro (1987) recommended a more complete understanding of the "contributions of ecological and physiological processes to variation in animal tissue nitrogen isotope ratios" (p. 201). Metabolic factors, such as urine concentration and urea output, may affect $\delta^{15}N$ values in water-conserving animals, yielding a pattern of results that would parallel those found in water-stressed animals living in arid environments. In their examination of marine and terrestrial food resources from South Africa, Sealy and colleagues (1987) noted that the $\delta^{15}N$ values of terrestrial animals varied widely with rainfall. They suggested that the regulation of urea excretion in response to water stress provides on explanation for this variation: by increasing excretion of isotopically light urea, animals can reduce the volume of urinary output, which thereby enriches the body of the animal in the heavier isotope.

Using both stable carbon and nitrogen isotopes, Ambrose and DeNiro (1989) tested the hypothesis that $\delta^{13}C$ and $\delta^{15}N$ values are influenced not only by diet, but also by climate and microhabitat. Analysis of bone and tooth samples from the central Rift Valley of Kenya and isotopic comparisons with modern herbivore collagen provided a means for tracing climatic changes during the Holocene. They concluded that, when diagenetic factors can be discounted and when adequate modern comparative data are available, stable isotope ratios of bone and tooth collagen can be used as crude indicators of prehistoric climate and habitat conditions. Van der Merwe (1989) showed that, by determining the $\delta^{13}C$ values for marker animal species such as pure browsers and grazers, researchers can obtain significant botanical information about ancient environments. In a recent study, Lee-Thorp and Beaumont (1995) used $\delta^{13}C$ values to evaluate season of rainfall in South Africa. The present distribution of C_3 and C_4 plants in South Africa is closely linked to season of rainfall: C_4 grasses are restricted to the interior and eastern regions where a summer rainfall regime prevails and C_3 grasses are confined to the winter rainfall zone in the western Cape Province and to the cool, high-altitude regions of the summer rainfall area. Their data indicated that $^{13}C/^{12}C$ ratios can be used to determine season of rainfall in South Africa based on the predominance of C_3 and C_4 plants in certain regions.

Trophic level effects on carbon and nitrogen isotopes

Every step in the food chain may be considered a trophic level. At each transfer to the next trophic level, a large portion of available energy is lost as heat because most of the fuel obtained in food is burned to do the everyday work of staying alive. Humans,

who occupy a position at the end of the food chain, may consume foods from a low trophic level (e.g. grains), which represents an efficient use of potential food energy, or they may consume food from a higher trophic level (e.g. beef). Unidirectional changes in stable isotope ratios along the continuum from primary producers to herbivores to primary carnivores to secondary carnivores are referred to as trophic level effects (Schoeninger, 1985).

Researchers studying the ecology of foodwebs have noted small increases in $\delta^{13}C$ and $\delta^{15}N$ values with successive trophic levels (DeNiro and Epstein, 1981; Lidén, 1995; Schoeninger, 1985; Schoeninger et al., 1983; Tuross et al., 1994). Analyses of $^{15}N/^{14}N$ ratios have consistently shown a stepwise enrichment of approximately 3‰ with each successively higher trophic level (Schoeninger, 1985; Schoeninger and DeNiro, 1984; Tuross et al., 1994; see Katzenberg and Kelly, 1991 and Lidén, 1995). As such, photosynthesizing plants have $\delta^{15}N$ values of 3‰ as compared with that of atmospheric nitrogen, which is 0‰. Similarly, those organisms subsisting entirely on plants will have $\delta^{15}N$ values of 6‰, with an enrichment in $\delta^{15}N$ values by 3‰ with each consecutive trophic level increase. The significance of this trophic level effect is most evident in a comparison of marine and terrestrial food chains: since marine food chains are much longer than terrestrial food chains, the top predator in a marine environment will have a higher $\delta^{15}N$ value than the top predator in a terrestrial system (Lidén, 1995). Results from carbon isotope studies are not nearly as uniform and notable changes in ^{13}C content with trophic level differences have not been noticed (Schoeninger, 1985; Vogel, as cited in van der Merwe, 1982). Accordingly, the vast amount of research on trophic level effects have focused on stable nitrogen isotopes.

In a study by Ambrose and DeNiro (1986), the $\delta^{15}N$ values of prehistoric and historic human populations from Africa were used to discriminate between diets with different protein sources: higher average $\delta^{15}N$ values were consistent with high trophic level sources of dietary protein. Katzenberg and Kelley (1991) looked at carbon and nitrogen isotope ratios in human skeletal remains from New Mexico spanning 500 years. They attributed the observed variation in $\delta^{15}N$ values to variations in the importance of meat as a dietary protein source.

Nitrogen isotopes and the determination of weaning age

Recently, there has been a growing body of literature added to studies of trophic level differences in $^{15}N/^{14}N$ ratios. This innovative research has been directed at determining if nursing infants should show enriched $\delta^{15}N$ values that reflect their higher trophic level position relative to their lactating mothers.

Using fingernail samples from contemporary mothers and infants, Fogel and co-workers (1989) showed a ^{15}N enrichment in infants of almost 3‰, which is consistent with a difference of one trophic level. They replicated their results using a pre-horticultural skeletal sample from Tennessee, which also indicated an enrichment in ^{15}N of collagen from one-year old infants. Katzenberg (1993) found a similar $\delta^{15}N$ enrichment in infants from three archaeological sites in Southern Ontario. Data from

each site suggested a trophic level shift from mother to nursing infant, reflecting the fact that the infants were obtaining their protein from their mother. In a subsequent examination of weaning age in a nineteenth century skeletal sample from Ontario, Katzenberg and Pfeiffer (1995) found a marked elevation in $\delta^{15}N$ values in infants relative to older children and adults. Their data also suggested a weaning age of just less than one year, which corresponds with the historic information available for the time period. In a recent study, Schurr (1997) used stable nitrogen ratios to determine the age at weaning, as well as the relationship between weaning and infant mortality for a prehistoric site in Indiana.

Nitrogen isotopes and the detection of disease

In a recent study of Nubian female mummies, White and Armelagos (1997) found elevated $\delta^{15}N$ values in females suffering from osteopenia, a degenerative bone disease that results in a loss of critical bone mass. These researchers have suggested that altered renal function in osteopenic females may affect urea nitrogen levels and modify renal processing of calcium and phosphorus, resulting in higher $\delta^{15}N$ values. Accordingly, the ^{15}N enrichment in females suffering from osteopenia may signify a relationship between physiological factors and nitrogen isotope ratios and $\delta^{15}N$ values may serve as useful isotopic markers for osteopenia in human skeletons (White and Armelagos, 1997).

Methods used in this study

The skeletal population used in this study originates from the late third millennium B.C. site of Tell Leilan in northeastern Syria (Figure 3.2). Human skeletal remains were recovered from approximately 47 different burials between 1979 and 1989 by a team of archaeologists from Yale University. Both articulated and disarticulated skeletons were recovered from burials in several different soundings throughout the site and accompanying grave goods ranged from simple pottery to ornamental goods made of copper and bronze (Chimko, 1997; Stein, 1990). Preliminary osteological analyses conducted at the University of Alberta indicated the presence of adult and subadult human specimens in the skeletal collection, as well as an assortment of domestic animal bones.

The human remains from these burials have been dated by stratigraphic analysis and have been assigned to particular periods in the Leilan chronology (summarized in Table 2.1). The skeletal samples used in this study originate from 16 different burials, dating from 2900 B.C. to approximately 1900 B.C. (see Table 3.1). The condition of the skeletal material ranged from clean with little flaking of cortical bone to highly weathered and damaged due to chemical contaminants such as consolidation preparations. Specimens that appeared to have been treated with preservatives or cleaned with chemical solvents were excluded from analysis. Skeletal specimens that appeared crumbly or that were filled with sediment were also excluded from analysis, since this sort of bone is unlikely to provide pure collagen (Ambrose, 1993). Only human rib samples that were

relatively clean and showed little cortical damage were used in this study. The samples discussed here comprise 16 different burials and represent 16 different individuals.

Bone collagen for isotopic measurements was extracted from these specimens following the procedure of Sealy (1986; see also Sealy and van der Merwe, 1986), since this method works well for older, more degraded specimens and produces "good gelatin" and collagen pseudomorphs with an amino acid profile similar to that of pure collagen (Schoeninger et al., 1989). All of the human rib samples were cleaned ultrasonically with distilled water to remove dirt and other environmental debris. The skeletal material was then air-dried and weighed. Only samples of dry bone weighing at least 1.0g were used in this analysis. Bone collagen was extracted using whole bone by soaking each rib fragment in 1% hydrochloric acid until the mineral fraction was dissolved, leaving only the organic portion ("collagen"). The demineralization process was considered complete once the bone appeared relatively translucent and flexible, which took anywhere from two weeks to almost two months. The samples were rinsed to neutrality with distilled water and frozen.

Due to laboratory constraints at the University of Alberta, the collagen samples were taken to the University of Calgary for analysis. The samples were freeze-dried for 24 to 48 hours. The freeze-dried collagen samples were weighed and the collagen yield for each sample was calculated. Approximately 1 mg of freeze-dried collagen was loaded into small tin containers, which were then loaded into an automatic sampler. The freeze-dried samples were then dropped into a Carlo-Erba gas analyzer and flash combusted to produce CO_2 and N_2 gas. The gases were separated and introduced into a Finnegan-Mat Tracer MAT mass spectrometer. The ratio of the stable carbon and nitrogen isotopes in the samples and in the internal lab standard[2] were recorded, relative to the international standards for carbon and nitrogen. C/N ratios were determined for each samples using the Carlo-Erba gas analyzer housed in the Department of Physics at the University of Calgary, under the direction of H.R. Krouse.

[4] The internal lab standard used at the University of Calgary is urea.

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Table 3.1. Archaeological information for burials used in this study

Burial Number	Leilan Period	Date (ca. B.C.)	Biological Age	Sex
L79 op1 108 B.2 [3]	IIIa	2900-2800	adult	unknown
L80 op1 61 Strat. 19	IIIc	2700-2600	adult	male
L80 op2 33/36 B.1	II	2400-2200	adult	female
L85 op4A 18 B.3	II	2400-2200	adult	male
L85 op4A 18 B.4	II	2400-2200	adult	unknown
L89 76 E20 B.1 area 4	II	2400-2200	infant	unknown
TL89 76 E20 B.2	II	2400-2200	subadult	unknown
L89 76 E20 B.3	II	2400-2200	infant	unknown
L89 76 E20 B.6	II	2400-2200	adult	unknown
L89 76 F20 rm 6 B.7	II	2400-2200	adult	unknown
L89 76 F20 B.5/9	II	2400-2200	adult	unknown
TL87 77 G01 B.1	II	2300-2200	adult	unknown
L87 77 G01 B.2	II	2300-2200	subadult	female
L87 77 G01 B.3	II	2300-2200	subadult	male
L87 57 FG05 65 (346)	I	1900-1728	infant	unknown
L87 57 FG05 64 B.2	I	1900-1728	infant	unknown

[5] L79 refers to the year of excavation at Tell Leilan; op1 refers to the operation number; 108 signifies the lot number; and B.2 refers to the burial number. Other information may refer to the grid square number or the plan number for that burial.

Figure 3.1. Distribution of stable carbon and nitrogen isotopes in marine and terrestrial systems (modified from Ambrose, 1993)

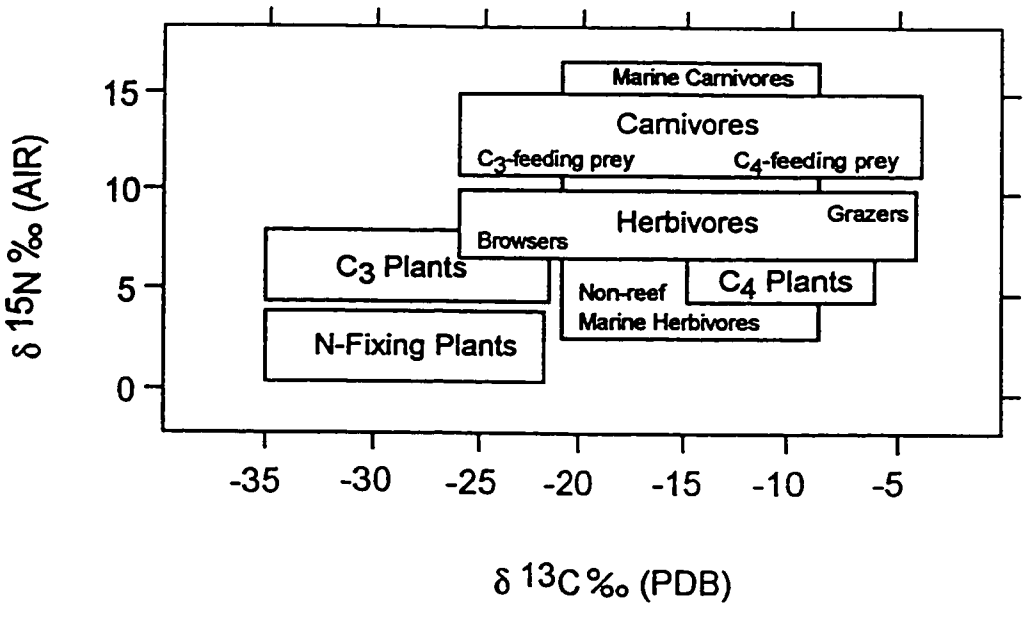
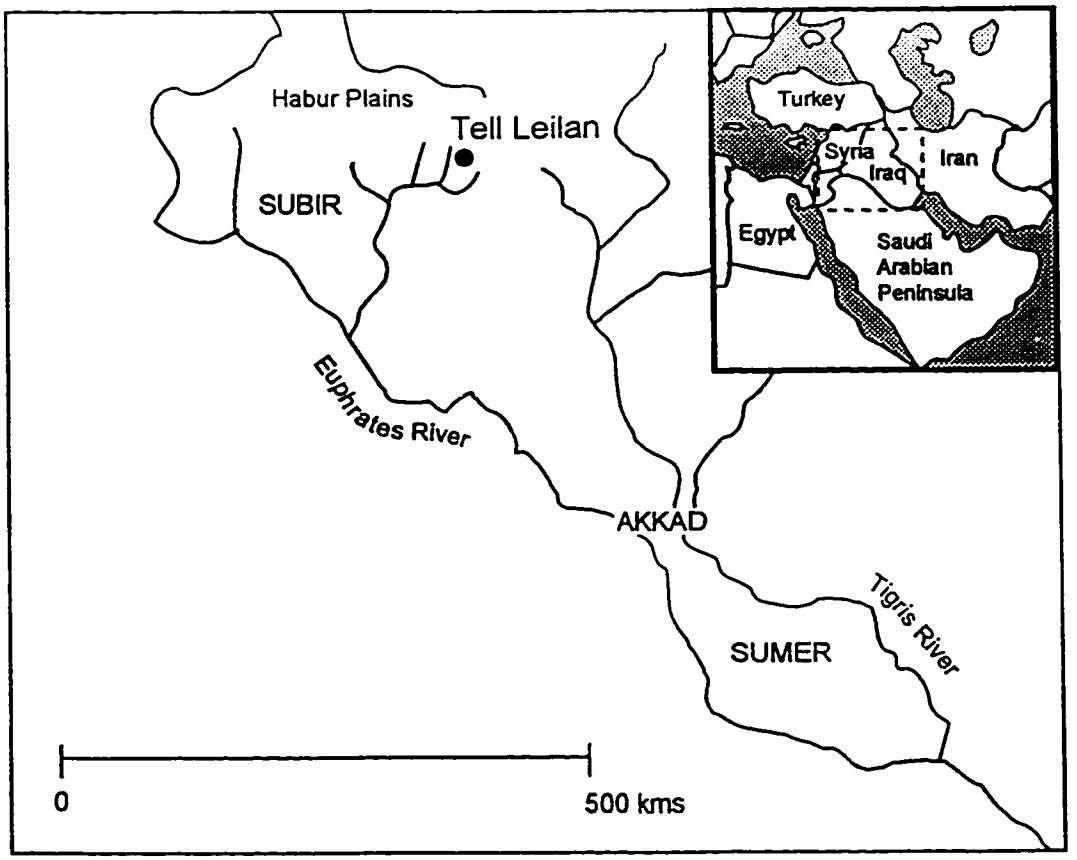


Figure 3.2. Map of ancient Mesopotamia showing the site of Tell Leilan (adapted from Gibbons, 1993)



Chapter 4

Tests of sample preparation methods and their effects on stable isotope values

Introduction

Anthropologists and archaeologists have devoted a significant amount of research time to the problem of assessing the preservation of prehistoric bone (see, for example, Hanson and Buikstra, 1987; Pate et al., 1989; Schoeninger et al., 1989). For researchers concerned with dietary and environmental reconstructions in past populations, the biological integrity of skeletal samples is paramount: any interpretation of past lifeways is predicated on the assumption that the chemical properties of excavated bone reflect the chemical makeup of the living individual. The biological signal in the carbon and nitrogen isotopes of fossil bone must be preserved in order to interpret the dietary patterns of ancient peoples.

The isolation of collagen from prehistoric human bones requires both precision and patience. Weathered skeletal material and fragmentary remains can present unique difficulties for a skeletal biologist concerned with the isolation of relatively pure collagen samples. This is particularly true for aged and degraded bone samples that may have been subjected to complex post-burial conditions and a variety of post-excavation preservatives. Accordingly, the chemical analysis of very old bone can be difficult at best. Sample specimens must be carefully chosen and cleaned and the process of bone demineralization must follow a cautious course designed to ensure the chemical integrity of collagen samples.

Scientists have studied the integrity of collagen in prehistoric bone by evaluating the efficacy of various collagen extraction techniques and by analyzing the precise chemical properties of pure bone collagen (see Tuross et al., 1988). In an examination of postmortem preservation of collagen isotope ratios, DeNiro (1985) determined that fossil bone samples with carbon to nitrogen ratios between 2.9 and 3.6 have biological integrity and have not been significantly altered by diagenetic processes. Sample purification techniques have also been evaluated in relation to humate contamination of fossil bone. The most commonly cited technique involves the treatment of demineralized bone with a weak solution of sodium hydroxide (see, for example, Ambrose, 1990; Chisholm et al., 1983). This approach produces an organic residue that is derived mostly from collagenous proteins, but with a slightly lower collagen yield than untreated samples (Ambrose, 1990; Katzenberg, 1989; Katzenberg et al., 1995). Chromatographic removal of humates produces the purest collagen residues and eliminates the problem of sample loss (Stafford et al., 1988). However, this technique is time consuming, expensive, and demands sophisticated laboratory equipment (Ambrose, 1990).

The likelihood of recovering biologically determined isotopic signals from archaeological bone has also been discussed with respect to the demineralization process itself. After the inorganic fraction has been removed, the chemical integrity of the collagen residue can be confirmed with amino acid analysis, since bone collagen has a unique amino acid profile. In an examination of the usefulness of two different demineralization methods, Schoeninger and colleagues (1989) suggested that collagen residues prepared using whole

bone chunks (as described by Sealy, 1986), rather than bone powder, had an amino acid profile more similar to that of pure collagen. Based on amino acid analysis, these researchers concluded that the demineralization of whole bone pieces in weak hydrochloric acid is the best way to obtain collagen samples ('pseudomorphs') with biological integrity and that C/N ratios may be less useful indicators of 'pure' collagen (Schoeninger et al., 1989).

The effect of acid concentration on $\delta^{13}C$ and $\delta^{15}N$ values

The skeletal material used in this study ranged in age from 3,700 years to almost 5,000 years old. As such, bone preservation varied from quite good to rather poor. Consolidation preparations were applied by the excavators in the field to numerous skeletal samples and preserving agents had stained the outer cortical layers of some bones. Only rib samples that were relatively well preserved, with little cortical wear, and without any obvious addition of preservatives were chosen for isotopic analysis. Cleaned whole bone chunks, approximately 1 cm in length, were demineralized in either 1% or 2% hydrochloric acid, following the procedure of Sealy (1986; see also Sealy and van der Merwe, 1986), as recommended by Schoeninger and colleagues (1989). Based on previous research (Moore et al., 1989; Schoeninger et al., 1989), it was assumed that a weakly acidic solution would yield the best collagen residues and would result in pseudomorph samples with chemical integrity. In order to test this assumption, 10 rib samples from one individual (*L80 op1 61 B.1*) were demineralized in 1% to 10% hydrochloric acid to determine the effect of acid concentration on the isolation of bone collagen.

Isotopic results indicated that acid concentration had no appreciable effect on $\delta^{15}N$ and $\delta^{13}C$ values (see Table 4.1, Figure 4.1, and Figure 4.2). In the case of carbon isotope ratios, there was significant overlap between 6 of the 10 $\delta^{13}C$ values and the other 4 ratios were within 2σ. Although there was slightly more variation in the nitrogen data, there was no trend observed with respect to acid strength and 6 of the 10 $\delta^{15}N$ values were essentially identical.

The atomic C/N ratios of the samples, which fall between the acceptable range of 2.9 to 3.6, indicate that the bone samples were not subjected to significant diagenetic alteration (DeNiro, 1985). Ambrose (1993) has suggested that $\delta^{13}C$ and $\delta^{15}N$ values do not differ with strong and weak HCl treatments when collagen is well-preserved. Although the isotopic ratios of the collagen samples in this study did vary, there was no directional change associated with acid strength. The observed variation in this set of samples may reflect slight contamination by lipids, carbonates, or humic acids in samples with C/N ratios over 3.4 (Ambrose, 1993). Additionally, the variation may be attributed to inconsistent mass spectrometric analysis, which may have resulted in shifts in data.

Although acid strength did not appear to affect isotopic ratios, it should be noted that the demineralization process was most satisfactory when 1% HCl was used. The collagen pseudomorphs prepared using 1% HCl tended to appear relatively translucent and flexible, while the collagen prepared using a stronger acid solution was frequently quite flaky in appearance. Demineralization in weak acid has been recommended by several researchers (e.g. Ambrose, 1990; Chisholm et al., 1983; Schoeninger et al., 1989) and experiments have

demonstrated that higher concentrations of collagenous proteins are obtained using weak acid treatments (Ambrose, 1990). Based on the experimental routine followed in this study, 1% HCl is recommended for aged bone samples where collagen preservation is questionable.

Collagen yield and the utility of treating aged or poorly preserved skeletal material with sodium hydroxide

Humic substances have an isotopic composition that reflects the plant biomass of the burial environment and can thereby confound isotopic dietary signals (Ambrose, 1993). Accordingly, the methodology for this study was to have included a sodium hydroxide soak to remove humic contaminants. During the initial preparation of pseudomorph samples, the collagen residue isolated from 15 different individuals (Table 4.2) was soaked in 0.125*M* NaOH for approximately 18 hours. However, the NaOH solution dissolved all of the collagen 'pseudomorph' samples, leaving only a clear, pale-coloured liquid. Even after repeated efforts, the base-insoluble collagen could not be chemically precipitated or centrifuged out of the solution. The entire set of samples was discarded and the subsequent isotopic analyses were performed on freshly prepared collagen pseudomorphs, omitting the NaOH soak.

There are several possible explanations for the failed NaOH wash. As with any laboratory experiment, human error may explain the problems encountered. Alternatively, the demineralized pseudomorph samples might have been composed of mostly non-collagenous proteins, which could have been dissolved by the NaOH. The most likely explanation, however, is that there was an insufficient yield of collagen. Several researchers have noted that NaOH treatments can result in a loss of collagen (Ambrose, 1990; Chisholm, 1989; Katzenberg, 1989; Katzenberg et al., 1995). If collagen yields are low, it follows that protein loss from a NaOH soak could significantly affect collagen residues and result in substantial sample loss. Since the pseudomorphs 'disappeared' in the NaOH soak before collagen yields could be calculated, the amount of collagen retained in the demineralized residues could not be determined. However, yields were calculated for collagen isolated from some of the same individuals at a later date (see Table 4.3). The extreme variation in the collagen yields, not only between samples but within repeats from the same individual, suggests poor collagen preservation. Given that roughly 50% of the samples produced unsatisfactory collagen yields, it is entirely likely that poor collagen yield was a factor in the NaOH destruction of the pseudomorphs.

Based on the severe difficulties encountered during the initial sample preparation, the NaOH soak was eliminated from subsequent collagen preparations. Because the use of NaOH serves to exclude non-collagenous organic material from isotopic analysis, it is typically regarded as an important step in the isolation of collagen from archaeological bone (Ambrose, 1990; Katzenberg et al, 1995). Nevertheless, it may be possible to determine the quantitative effects of humic substances on isotopic ratios when a NaOH soak is not used. Katzenberg and colleagues (1995) demonstrated that the inclusion of humic substances in isotopic analysis results in $\delta^{13}C$ values that are slightly more negative (on the order of 0.24‰), although the extent to which carbon ratios are affected depends, in large part, on the type of burial environment (Ambrose, 1990). In the same study (Katzenberg et al., 1995), the

inclusion of humic contaminants resulted in $\delta^{15}N$ values that were, on average, 0.16‰ higher. Since these are within the precision of analysis, it appears that the exclusion of a NaOH soak will affect dietary interpretations only slightly (Katzenberg et al., 1995).

Measurement variation

Multiple rib samples were demineralized for several burials[b] in order to test the integrity of the bone samples and to examine the accuracy of mass spectrometric measurements. Thirty-one pseudomorph samples from 16 different individuals were analyzed with the mass spectrometer at the University of Calgary.

As is clear from the data presented in Table 4.4, there was significant variation in $\delta^{13}C$ and $\delta^{15}N$ values between replicates. In some instances, the isotopic variation between samples from the same individual approached 1.2‰ for carbon and 1.3‰ for nitrogen. Mass spectrometric precision at the University of Calgary is ± 0.4‰ for carbon and ± 0.3‰ for nitrogen (M.A. Katzenberg, pers. comm.). The precision of analysis is typically ± 0.1‰ or less for carbon and ± 0.2‰ or less for nitrogen (Ambrose, 1993). Since the level of measurement precision in this study is less than idael, it may therefore account for some of the observed variation in repeated measurements for the same individual. Varying degrees of contamination by humic substances, normally removed by a NaOH wash, may also account for intraindividual variation, since humic contaminants may alter $\delta^{13}C$ and $\delta^{15}N$ values (Katzenberg et al., 1995).

Conclusions

The methodology followed in this study takes into account the age and degraded nature of many of the sample specimens. A weak acid solution proved to be preferable for obtaining collagen pseudomorphs that were elastic and translucent. Due to time constraints and laboratory limitations, an in-depth analysis of the significance of the NaOH effects on the skeletal material could not be conducted. The expense associated with amino acid screenings, for example, precluded the analysis of the precise chemical makeup of the collagen samples.

The analysis of pseudomorph replicates revealed significant variation in $\delta^{13}C$ and $\delta^{15}N$ values for some individuals. This cannot be attributed to interelement variation within the human skeleton, however, since sample selection was designed to eliminate potential variation by selecting only rib fragments for isotopic analysis. Additionally, every effort was made to ensure the proper demineralization of all bone samples: only dilute (1%) HCl was used and the samples were not removed from the acid until they appeared translucent and flexible (Sealy, 1986; Schoeninger et al., 1989). Some of the variation between collagen replicates may be explained by burial conditions. Unfortunately, the mortuary and

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[b] Although several rib fragments were originally taken from each burial, pseudomorph replicates for each burial were not available at the end of the acid treatment. Several samples had to be discarded due to human error and improper demineralization.

archaeological reports for Tell Leilan provide only cursory information on provenience and distribution of bone fragments at burial sites. The variation may also be explained, at least in part, by a poorer level of mass spectrometric precision than is usually considered ideal, and this is currenly being investigated. Pseudomorph samples from 12 burials are being run in triplicate with a blank reference between each individual in order to control for any 'memory effect' present in the mass spectrometer that may confound isotopic ratios.

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Table 4.1 - Acid concentration and stable isotope ratios

Acid Concentration	$\delta^{13}C$ (‰ $_{PDB}$)	$\delta^{15}N$ (‰ $_{AIR}$)	Carbon to Nitrogen Ratio
1%	-18.8	7.8	3.18
2%	-19.0	7.8	3.34
3%	-19.1	8.0	3.37
4%	-18.5	8.1	3.39
5%	-20.0	7.5	3.44
6%	-18.0	7.8	3.37
7%	-19.0	7.7	3.32
8%	-18.8	8.3	3.46
9%	-18.9	7.7	3.23
10%	-18.6	7.8	3.32

Table 4.2 - Burials used for the NaOH soak

Burial Number	Date (ca. B.C.)	Biological Age	Sex
L79 op1 108 B.1	2900-2800	adult	unknown
L80 op1 61 Strat.19	2700-2600	adult	male
L80 op2 33/36 B.1	2400-2200	adult	female
*L85 op4A 18 B.1**	2400-2200	infant	unknown
L85 op4A 18 B.3	2400-2200	adult	male
L85 op4A 18 B.4	2400-2200	adult	unknown
*L89 76 E20 B.1 area 4**	2400-2200	infant	unknown
TL89 76 E20 B.2†	2400-2200	subadult	unknown
L89 76 E20 B.6	2400-2200	adult	unknown
L89 76 F20 rm.6 B.7	2400-2200	adult	unknown
L89 76 F20 B.5/9	2400-2200	adult	unknown
TL87 77 G01 B.1	2300-2200	adult	unknown
L87 77 G01 B.2†	2300-2200	subadult	female
L87 57 FG05 65 (346)‡	1900-1728	infant	unknown
L87 57 FG05 64 B.2‡	1900-1728	infant	unknown

* infant 6 months or older
† approximately 15 years of age
‡ infant under 6 months of age

Table 4.3 - Collagen yields obtained for second group of samples prepared after the loss of a first group of samples in a NaOH soak

Burial Number	Collagen Yield[7]
L79 op1 108 B.1	7-12%
L80 op1 61 Strat.19	4%
L80 op2 33/36 B.1	0%
L85 op4A 18 B.3	9-15%
L86 op4A 18 B.4	1-8%
TL89 76 E20 B.2	2%
L89 76 E20 B.6	5-9%
L89 76 E20 rm6 B.7	9%
L87 77 G01 B.2	2%
L87 57 FG05 64 B.2	17%

[7] A range of values indicates that collagen yeilds were calculated for more than one sample from the same burial (see Measurement Variation).

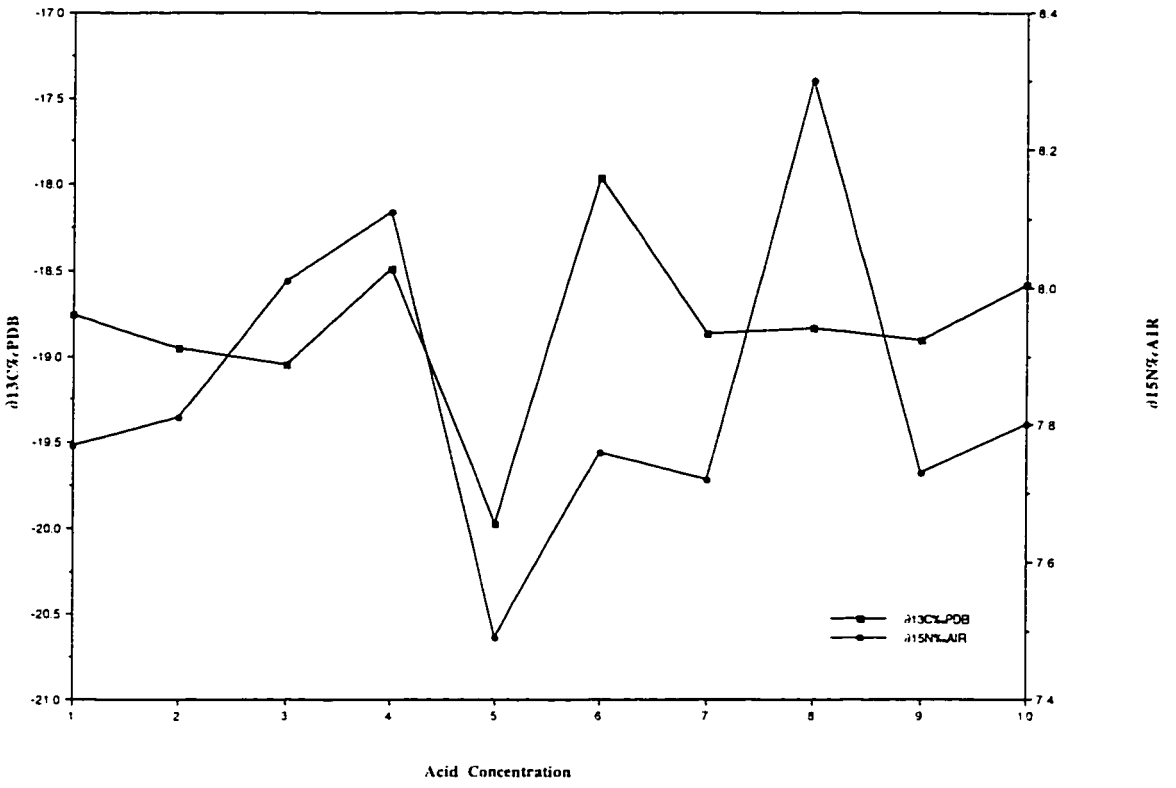
Table 4.4. Measurement variation: results of isotopic analysis of replicate rib samples

Burial Number	Sample Number	$\delta^{13}C$ (‰ PDB)	$\delta^{15}N$ (‰ AIR)
L79 op1 108 B.1	a	-20.4	7.7
	b	-19.2	8.1
	c	-19.1	7.3
L80 op1 61 Strat.19	a	-18.8	8.1
	b	-19.4	8.7
L80 op2 33/36 B.1	a	-19.8	8.8
	b	-20.0	8.7
L85 op4A 18 B.3	a	-19.6	8.9
	b	-20.5	9.3
	c	-19.4	9.3
	d	-19.2	8.3
L85 op4A 18 B.4	a	-19.2	9.0
	b	-19.1	9.1
*L89 76 E20 B.1 area 4**	a	-19.3	9.2
TL98 76 E20 B.2	a	-18.8	10.3
	b	-19.2	9.5
*L89 76 E20 B.3**	a	-20.3	7.6
L89 76 E20 B.6	a	-19.1	10.2
	b	-19.2	10.2
	c	-19.3	8.9
L89 76 F20 rm.6 B.7	a	-19.0	9.6
	b	-18.9	8.5
L89 76 E20 B.5/9	a	-19.9	9.5
	b	-19.6	9.2
	c	-19.3	8.4
TL87 77 G01 B.1	a	-21.1	8.7
L87 77 G01 B.2	a	-18.4	10.5
L87 77 G01 B.3	a	-19.0	8.1
L87 57 FG05 65 (346)‡	a	-18.9	9.1
L87 57 FG05 64 B.2‡	a	-18.2	12.0
	b	-18.1	10.7

* infant under 6 months of age

‡ infant 6 months or older

Figure 4.1 - The effect of acid concentration on $\delta^{13}C$ and $\delta^{15}N$ values



Chapter 5

Stable isotope evidence for dietary patterns and environmental conditions at Tell Leilan, Syria, ca. 2900-1900 B.C.

Introduction

By understanding prehistoric diet, anthropologists can interpret the modes of adaptation in ancient human groups. Accordingly, researchers are able trace the ancient lifeways of our species and determine the environmental, cultural, and physiological significance of dietary patterns over time. Dietary research has the potential to contribute greatly to our understanding of prehistoric ecological conditions, cultural practices, social divisions, and health. The isotopic analysis of bone collagen has proven particularly useful for interpreting prehistoric patterns of food consumption.

Isotopic analyses, however, have largely been limited to the study of skeletal remains from the Americas, Europe, and Sub-Saharan Africa, where well-preserved skeletal material is readily accessible from museum collections or archaeological sites. The dry burial conditions, not to mention political unrest and uncertainty, have resulted in only minor contributions to stable isotope research from the Middle East and northern Africa (see Iacumin et al., 1996 and White and Schwarcz, 1989, 1994 for exceptions). However, many of the world's first civilizations are thought to have arisen in this desert region, particularly in ancient Mesopotamia, in what is now Syria and Iraq. As such, the potential significance of stable isotope research in the Near East is great. In the case of Mesopotamia, the reconstruction of dietary adaptations has the potential to extend our capacity for understanding the origins of agriculture and early urban life.

The middle of the third millennium B.C. marks the pinnacle of Mesopotamian urbanization and growth (Adams 1981; Weiss, 1986). This urban expansion, while initially centred in the southern empire of Akkad, extended to northern Mesopotamia and southern power was expanded to include the rain-fed agricultural regions of the north. The site of Tell Leilan in northeastern Syria (Figure 5.1) has provided much of the information for researchers attempting to understand the mechanisms of early urban expansion in northern Mesopotamia and has supplied anthropologists with a wealth of information on the rise and fall of early city states.

The objectives of this study are (1) to identify the general dietary patterns of an ancient Mesopotamian population from Tell Leilan, Syria; (2) to chronicle any shifts in dietary adaptations during the rise and fall of this early city; and (3) to use the stable isotope data to interpret possible environmental changes during the time period being investigated.

Background

Stable Isotope Analysis

The first palaeodietary reconstructions using stable isotopes utilized carbon isotope ratios to trace the introduction of maize in the northeastern United States (van der Merwe and Vogel, 1978; Vogel and van der Merwe, 1977). The practical scope of palaeodietary analysis was later expanded to include the use of stable carbon and nitrogen isotopes in estimations of human dependence on animal versus plant protein (Ambrose, 1986; Ambrose and DeNiro, 1986; Schwarcz et al., 1985) and calculations of the dietary importance of marine and terrestrial foods (Chisholm et al., 1982; Larsen et al., 1992; Lovell et al., 1986a; Mays, 1997; McGovern-Wilson and Quinn, 1996; Pate, 1997; Schoeninger et al., 1983; Schoeninger and DeNiro, 1984; Sealy and van der Merwe, 1988; Tauber, 1981; Walker and DeNiro, 1986). Trophic level effects on carbon and nitrogen isotope ratios have also been investigated (DeNiro and Epstein, 1981; Katzenberg and Kelley, 1991; Lidén, 1995; Schoeninger, 1985; Tuross et al., 1994) and nitrogen isotopes have been used to determine weaning age (Fogel et al., 1989; Katzenberg, 1993; Katzenberg and Pfeiffer, 1995; Katzenberg et al., 1996; Katzenberg et al., 1993; Schurr, 1997; White and Schwarcz, 1994). The potential of nitrogen isotope ratios as markers of disease has also been contemplated (White and Armelagos, 1997) and significant progress has been made in understanding the effects of diagenesis on bone (e.g. Ambrose, 1990; DeNiro, 1985; Schoeninger et al., 1989; and Tuross et al., 1988). Summaries of these and other advances in stable isotope research have been compiled in several recent reviews (Ambrose, 1993; Chisholm, 1989; Katzenberg, 1992; Keegan, 1989; Schoeninger and Moore, 1992; Schwarcz and Schoeninger, 1991).

The use of stable isotope ratios in palaeodietary reconstructions is predicated on the observation that different dietary resources have different and distinct isotopic make-ups. In the case of carbon, it is possible to distinguish between different groups of plants that assimilate carbon at different rates. C_3 plants, such as wheat and rice, have average $\delta^{13}C$ values of approximately -27 ‰, while C_4 plants such as maize and sugarcane have average $\delta^{13}C$ values of about -11 ‰ (Raven et al., 1986). Nitrogen isotope variation is detectable in both marine and terrestrial foodwebs. The $\delta^{15}N$ values of terrestrial plants are approximately 4‰ lower than marine plants and plants grown in cool, moist forest soils have lower $\delta^{15}N$ values than plants grown in dry savanna or desert soils (Ambrose, 1993). Trophic level shifts in nitrogen ratios are also evident, with herbivores showing an increase of approximately 3‰ from the plants that they consume (Katzenberg, 1992) and carnivores showing an additional increase of about 3‰ over herbivores (Schoeninger and DeNiro, 1984).

In order to reconstruct the diet of past peoples, it is necessary to examine how the isotopic ratios of dietary plants and animals are affected by human metabolism. During

the course of metabolism and collagen formation, the isotopic ratios of food resources may be altered before the food energy is stored in human tissue (Ambrose and Norr, 1993; Keegan, 1989; van der Merwe, 1982). Early examinations of isotopic fractionation indicated that the $\delta^{13}C$ and $\delta^{15}N$ values in consumer tissues were enriched when compared with the isotopic composition of their diet (DeNiro and Epstein, 1978, 1981; van der Merwe and Vogel, 1978; Vogel and van der Merwe, 1977). Subsequent studies have suggested that carbon isotope ratios in human bone collagen are approximately 5‰ higher that dietary $\delta^{13}C$ values (see Ambrose, 1993 and Keegan, 1989 for reviews; see also van der Merwe,1982; Katzenberg et al., 1995; and Schoeninger, 1989). The diet-to-collagen enrichment factor for nitrogen, although calculated to be approximately 2.5 to 3.0‰ for humans (DeNiro and Epstein, 1981; Keegan, 1989), is less certain than that for carbon, since $\delta^{15}N$ values may also be influenced by climatic and physiological factors (Ambrose, 1993).

Palaeoenvironmental conditions can also be studied using stable isotope data and, in some instances, environmental change over time can be assessed. By reconstructing the general dietary patterns of an ancient humans and animals, growing conditions can be estimated and climatic trends can be considered (e.g. Ambrose and DeNiro, 1989; Lee-Thorp and Beaumont, 1995). This type of analysis has the potential to be particularly useful in reconstructing the diet and climatic history of abandoned civilizations and collapsed cities.

The Cultural Context of the Skeletal Sample

Archaeological data from the site of Tell Leilan have defined the origin, growth, and collapse of this once powerful city. The dry-farming urbanized landscape at and around Tell Leilan developed suddenly at approximately 2600 B.C. (Weiss and Courty, 1993). During the 200 years that followed, settlement patterns across the Habur Plains were completely reorganized and large, planned cities such as Tell Leilan were constructed throughout the region (Adams, 1981; Weiss et al., 1993). By about 2400 B.C., settlement numbers reached new heights and an urban hierarchy became firmly established. A defensive wall was constructed around the Leilan Acropolis and state power was consolidated in the cities across northern Mesopotamia (Weiss and Courty, 1993; Weiss et al., 1993). From approximately 2300 to 2200 B.C., agricultural production across the Habur Plains came under imperial control as the southern Mesopotamians, led by Sargon of Akkad, stormed the northern plains and extended their political power (Weiss, 1990; Weiss, 1996; Weiss et al., 1993). Under Akkadian rule, the northern centres continued to grow and the southern rulers benefited from imperial taxes and agricultural surplus. In terms of wealth and abundance, urban life had reached new heights in the Near East. This prosperity, however, was to last only about one hundred years. At approximately 2200 B.C., misfortune apparently befell the peoples of

Mesopotamia and the Akkadian empire collapsed (e.g. Issar, 1995; Rosen, 1995; Weiss, 1996; Yoffee, 1988). It was not until approximately 300 years later that settlement was reestablished in Mesopotamia and the abandoned city of Tell Leilan was revived as a new administrative centre for all of northern Mesopotamia (Weiss and Courty, 1993).

Materials and Methods

Human rib samples from 16 individuals from the site of Tell Leilan were used in this study. The biological age and sex of the specimens were determined by reference to standard osteological protocols (e.g. Bass, 1995) and their chronological positions were obtained from field reports prepared by Yale University archaeologists. Burial numbers, dates, and age and sex characteristics are presented in Table 5.1.

Skeletal samples were selected based on availability and apparent contamination: rib fragments that appeared to have been cleaned with chemical solvents or treated with preservatives were excluded from analysis. The ribs were cut into small pieces, about 1 cm^2, and the specimens were cleaned of dirt and debris using a soft brush and were then washed ultrasonically with distilled water. The samples were air-dried and weighed so that collagen yields could be determined later. Following the method described by Sealy (1986; see also Sealy and van der Merwe, 1986), the cleaned rib pieces were demineralized in 1% hydrochloric acid until they appeared translucent and flexible. This process took anywhere from two weeks to almost two months. This method has been endorsed for producing collagen residues with biological integrity and amino acid profiles similar to that of pure collagen (Schoeninger et al., 1989).

The resulting organic residues ('collagen pseudomorphs') were frozen and taken to the Department of Archaeology at the University of Calgary where the samples were freeze-dried for 24 to 48 hours. For each sample, approximately 1mg of lypholized collagen was weighed into small tin cups and dropped them into a Carlo-Erba gas analyzer. The samples were flash combusted to produce CO_2 and N_2 and the gases were then introduced into a Finnegan-Mat Tracer MAT mass spectrometer. The ratio of the stable carbon and nitrogen isotopes in the samples and the internal lab standard[8] were recorded relative to the international standards for carbon and nitrogen[9]. Carbon/nitrogen ratios were determined using the Carlo-Erba gas analyzer housed in the Department of Physics at the University of Calgary, under the supervision of H.R. Krouse.

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[8] The internal lab standard used at the University of Calgary is urea.

[9] The standard for carbon is a limestone fossil from the Peedee formation in South Carolina (PBD) and the standard for nitrogen is atmospheric air (AIR).

Results and Discussion

Carbon to Nitrogen Ratios

All of the samples discussed in this study were analyzed for carbon and nitrogen to determine the integrity of the collagen. The values, which have been converted to molar ratios, are reported in Table 5.2. While most of the C/N ratios fall within the range suggested by DeNiro (1985) as indicative of unaltered collagen, 9 of the 31 samples (29%) had higher ratios of carbon to nitrogen than the acceptable limit of 3.6. Since contamination of collagen samples with humic substances results in higher C/N ratios (Katzenberg, 1989), the removal of humic contaminants is typically included in isotopic studies. The protocol involves soaking demineralized bone collagen in dilute sodium hydroxide for approximately 20 hours (see Ambrose, 1993 and Katzenberg, 1992). Unfortunately, the NaOH solution apparently dissolved all of the collagen residues in the initially prepared samples and therefore the first set of samples was discarded and the subsequent isotopic analyses were performed on freshly prepared collagen pseudomorphs, omitting the NaOH soak. Since there is only a very small amount of nitrogen in humic contaminants, stable nitrogen isotopic ratios should not be affected by their presence (Katzenberg, 1989). There is, however, a significant amount of carbon in humic substances, and thus $\delta^{13}C$ values may be affected by plant residues that were present in the burial environment (Katzenberg, 1989). Therefore, some of the isotopic ratios obtained in this study may have altered by the presence of base soluble contaminants.

There is no evidence, however, for a systematic effect on isotopic ratios in this study. As shown in Table 5.2, the results of this study demonstrate no consistent isotopic trend associated with a sample's C/N ratio: some samples with C/N ratios over 3.6 exhibited lighter $\delta^{13}C$ values (e.g. *TL87 77 G01 B.1*), while others appeared to be unaffected by potential humic contaminants (e.g. *L85 op4A 18 B.3*). Accordingly, C/N ratios outside the commonly cited accepted limit of 3.6 were not considered to be justification for the exclusion of samples from further analysis.

Biological Age and Sex

The precise effect of biological age on stable carbon isotope ratios has not been studied extensively, despite the fact that nutritional requirements are largely a function of developmental stage (White and Schwarcz, 1994). In an early study of age effects, Lovell and colleagues (1986b) found that $\delta^{13}C$ values did not vary with age among prehistoric bison-hunters from Saskatchewan. A lack of an age effect was also found among Maya maize agriculturists (White and Schwarcz, 1989) and Nubian agricultural populations (White and Schwarcz, 1994). Conversely, Katzenberg (1993; see also Katzenberg et al.,

1993) noted significant differences in $\delta^{13}C$ values between young children (under two years) and adults in a southern Ontario agricultural population. These studies suggest that variations between age and carbon isotope ratios are not physiological, but reflect the dietary adaptations of particular groups. In this study, carbon isotope ratios were highest among infants under six months of age, with an average $\delta^{13}C$ value of -18.5‰ ± 0.6‰ (Figure 5.2). Infants over six months of age exhibited the lowest average $\delta^{13}C$ value of -19.8‰ ± 0.7‰. The higher carbon ratios for infants under six months may reflect a slight trophic level effect for carbon (+1‰) in nursing infants (Katzenberg, 1993). However, the small sample size for infants under six months of age and for infants over six months of age makes this hypothesis difficult to prove. Time is also a confounding variable in this study, since the two infants under six months of age are both from Period I. The slight variation in $\delta^{13}C$ values between age groups may also be a function of measurement precision.

Age effects on $\delta^{15}N$ values have been studied in depth (see Fogel et al., 1989 and Katzenberg et al., 1996) and researchers have recognized a $\delta^{15}N$ enrichment in breast-feeding infants. This enrichment, which is on the order of 3‰ (e.g. Fogel et al., 1989; Katzenberg and Pfeiffer, 1995), reflects the higher trophic level position of nursing infants relative to their mothers. Peak $\delta^{15}N$ values are observed in infants from several months after birth until just after the introduction of supplemental foods (Katzenberg et al., 1996), allowing for the calculation of a weaning age in archaeological populations if adequate skeletal material is available. The bone collagen data from Tell Leilan may suggest such a trophic level effect of weaning. Infants under six months of age showed enriched nitrogen isotope ratios, with an average $\delta^{15}N$ value of 10.2‰ ± 1.6‰ (Figure 5.3). Infants over six months of age exhibited the lowest nitrogen isotope ratios, with an average value of 8.4‰ ± 1.2‰. A potential weaning effect in this population is most evident in one infant: *L87 57 FG05 64 B.2* (Table 5.3). This infant exhibited the highest average $\delta^{15}N$ value (11.3‰ ± 0.9‰), which is 2.6‰ higher than the average $\delta^{15}N$ value for the one adult female in this study. This enrichment value coincides with the results from other isotopic studies that identified a weaning effect (see Katzenberg et al., 1996 for a review). Unfortunately, the extremely small sample size in this study precludes any conclusive assessment of the weaning process at Tell Leilan. As with $\delta^{13}C$, time may also be a confounding variable: all of the infants under six months of age were recovered from Period I deposits and there is no Period I adult female data available for comparison. As such, a weaning effect in this population cannot be fully assessed.

The effects of sex on stable carbon and nitrogen isotope ratios have been examined in both human (e.g. Lovell et al., 1986b) and animal populations (e.g. DeNiro and Schoeninger, 1983). Using controlled feeding experiments with minks and rabbits, DeNiro and Schoeninger (1983) determined that the differences in $\delta^{13}C$ and $\delta^{15}N$ between males and females on the same diet were insignificant, on the order of 1‰ or less. Lovell and co-workers (1986b) found similar uniformity in $\delta^{13}C$ for a population of prehistoric

plains hunter-gatherers, with differences of only 0.3‰ between the sexes. Based on these and other studies, researchers have generally concluded that isotopic differences between males and females which exceed the precision of measurement represent dietary differences (Chisholm, 1989; Lovell, unpublished manuscript; Schwarcz and Schoeninger, 1991). In this study, sex was only known for five of the skeletal specimens: two subadults from Period II, two adults from Period II, and one adult from Period III (Table 5.1). The $\delta^{15}N$ value for the subadult female (*L87 77 G01 B.2*) was notably higher (+2.5‰) than that for the subadult male (*L87 77 G01 B.3*), suggesting a potential dietary difference between the two individuals (Table 5.2 and Table 5.3). A higher consumption of meat protein might explain the elevated nitrogen value for the subadult female. There were no significant differences in carbon isotope ratios for these subadults. For the two Period II adults, the $\delta^{13}C$ values and $\delta^{15}N$ values were essentially the same: average carbon ratios differed by only 0.5‰ and average nitrogen values differed by only 0.07‰ (Table 5.3), which is close to or within the precision of analysis. The extremely small sample size in this study precludes any in depth analysis of sex effects on isotopic ratios. Therefore, dietary divisions along sexual lines, although possible given the results for the subadult samples, cannot be fully assessed at this time.

Stable Carbon Isotopes

Based on total samples analyzed, including replicates, $\delta^{13}C$ values in bone collagen ranged from -18.1‰ to -21.1‰, with a mean value of -19.3 ± 0.7‰ (Table 5.2). These carbon isotope ratios suggest a diet composed primarily of C_3 plants. Archaeobotanical analyses indicate that Leilan farmers relied heavily on emmer wheat, durum (*Triticum durum*), and barley throughout Periods III and II (Wetterstrom, n.d. a, b). This crop complex is composed entirely of C_3 plants, which have average $\delta^{13}C$ values of approximately -27‰ (Figure 5.4). Small-seeded wild C_3 plants, such as grasses and legumes, also contributed to the Leilan diet (Wetterstrom, n.d. b). By modern analogy, two C_3 plants dominate the agricultural landscape of the Habur Plains: wheat and barley (Ergenzinger et al., 1988).

Since there is a diet-to-collagen enrichment factor of approximately +5.1‰ for carbon (Schoeninger, 1989; van der Merwe, 1982), the dietary isotope ratios of C_3 plants would appear to account for the carbon isotope ratios observed in the Leilan bone collagen. However, this conclusion is based on the assumption that the $\delta^{13}C$ values of bone collagen are directly related to the isotopic composition of the diet. The actual isotopic signatures of bone collagen may deviate from this theory, since some dietary isotope labels may be preferentially preserved in bone collagen (Ambrose and Norr, 1993; Little and Little, 1997; Schwarcz, 1991). Although relatively little work has been done on the effect of isotopic "routing" in estimations of prehistoric diets, researchers have suggested that protein carbon contributes a greater fraction of carbon to bone

collagen than does carbohydrate carbon (Ambrose and Norr, 1993; Schwarcz, 1991). This sort of routing effect in carbon is best evaluated by testing the isotopic homogeneity between collagen and bone apatite: both tissue types should show similar isotopic compositions if carbon is being preferentially routed from certain dietary sources (Krueger and Sullivan, 1984; Schwarcz, 1991). Based on controlled feeding experiments, Ambrose and Norr (1991) found that animals subsisting on C_3 energy-rich or C_3 protein-rich diets had $\delta^{13}C$ values of approximately -20‰. Extensive isotopic analyses of various prehistoric humans with known diets have also indicated that individuals subsisting on a diet of C_3 plants, with or without C_3 meats, should have $\delta^{13}C$ values between -24‰ and -18‰ (Krueger and Sullivan, 1984). This range of values is consistent with the carbon isotope ratios in this study. Based on average values for the 16 Leilan individuals (Table 5.3), the carbon isotope ratios are quite homogeneous with $\delta^{13}C$ values clustering between -18.8‰ and -20.3‰ (Figure 5.5). One of the two outlying heavy carbon values (an infant under six months of age) may reflect a slight trophic level effect of weaning. The one light $\delta^{13}C$ value may indicate a migrant individual or may reflect measurement variation.

Stable Nitrogen Isotopes

Based on total samples analyzed, including replicates, $\delta^{15}N$ values ranged from 7.3‰ to 12‰, with a mean value of 9.1 ± 1.0‰ (Table 5.2). Nitrogen isotopic ratios were highest among infants under six months of age. When these infant data are excluded from analysis, the total range of $\delta^{15}N$ values is 7.3‰ to 10.5‰.

The remains of one pulse crop, lentil (*Lens culinaris*), suggest that this legume was an important cultigen during Periods III and II (Wetterstrom, n.d. b). Nitrogen-fixing terrestrial plants such as lentils exhibit $\delta^{15}N$ values of approximately 1‰ ± 2‰ (Price et al., 1985; see also Ambrose, 1993). The nitrogen fractionation factor between diet and bone collagen is approximately 3‰ (Schoeninger, 1989), resulting in bone collagen $\delta^{15}N$ values of approximately 4-5‰ for an individual subsisting entirely on N_2-fixing legumes. The bone collagen $\delta^{15}N$ values in this study are notably higher than this, with average $\delta^{15}N$ values ranging from 8.1‰ to 10.5‰[3] (Table 5.3).

Dry, desert-like soils, saline soils, and soils with significant inputs from animals (e.g. manure and urine) have higher nitrogen isotope ratios (Ambrose, 1993). Animal inputs would have likely contributed to nitrogen soil enrichment at Tell Leilan since animals apparently grazed extensively throughout pastures and fallow fields (Wetterstrom, n.d. b). Archaeological soil analyses have also indicated that salinity was a

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[10] Excluding average nitrogen isotope ratios of infants.

problem throughout Mesopotamia during the third millennium, particularly between 2400 and 2100 B.C. (Jacobsen, 1982).

Sheep, goats, and cattle constitute a substantial portion of the faunal assemblage available for Tell Leilan (M. MacKinnon, pers. comm.). Archaeological studies have revealed that Leilan farmers employed agricultural strategies aimed at minimizing the risk of lost crops during the dry years that frequently prevailed on the Habur Plains (Weiss, 1986; Wetterstrom, n.d. a). The farming of drought-tolerant browsers (caprines) would have further maximized potential food intake during dry spells. Drought-tolerant animals that excreted ^{15}N-depleted urea as an adaptive strategy would have higher $\delta^{15}N$ values than obligate drinkers (Ambrose, 1993). Water-conserving animals typically have nitrogen isotope ratios on the order of 8.3‰, whereas water-dependent species have lower values, ranging from 6.2‰ to 7.9‰ (White and Schwarcz, 1994). Accordingly, the dependence on caprine species as a protein source, combined with the consumption of low $\delta^{15}N$ legumes, might explain the nitrogen isotope ratios for Tell Leilan. Analysis of Leilan fauna is necessary to support this assumption, but adequate remains are as yet unavailable.

The variation in nitrogen isotope ratios in this study may be attributed to variations in the dietary importance of meat and protein-rich legumes. Botanical analyses have revealed a Period II difference in pulse crop abundance between a ration-worker's household and Acropolis residences (Wetterstrom, n.d. b). Although faunal analyses have not been undertaken, similar variations might be expected for dietary animal remains if certain households had preferential access to particular dietary items. Archaeological analyses of mortuary data have provided evidence of social stratification at Tell Leilan during Periods III and II (Schwartz, 1986; Stein, 1990), which may have resulted in preferential access to certain food items based on status or political position.

Unfortunately, the mortuary reports for Tell Leilan are extremely sketchy. The limited data available suggest the potential for social stratification during the mid-third millennium B.C., but the archaeological evidence is at present too sparse to establish a social hierarchy among the inhabitants of Tell Leilan. Based on average isotope ratios (Table 5.3), the nitrogen data are relatively homogeneous, with $\delta^{15}N$ values clustering between 7.6‰ and 9.9‰ (Figure 5.5). Desert sheep and goats and cattle, which may have been farmed at Tell Leilan (M MacKinnon, pers. comm.; Wetterstrom, n.d. a,b), have $\delta^{15}N$ values in the central Rift Valley of Kenya of 8.2‰, 8.5‰, and 6.2-7.9‰, respectively (Ambrose, 1991; White and Schwarcz, 1994). If these values can be used as a reference set, then consumption of these meats would result in $\delta^{15}N$ values in human bone collagen of approximately 10‰ to 12‰ (see White and Schwarcz, 1994). The additional consumption of N_2-fixing legumes, such as lentils, would result in slightly lower nitrogen values, which would coincide with the range of nitrogen values for Tell Leilan.

Dietary Change Over Time

Average $\delta^{13}C$ values during Leilan Periods III, II, and I are presented in Table 5.4 and Figure 5.6. During Period III, average $\delta^{13}C$ values ranged from -19.1‰ to -19.5‰, with an mean of -19.3‰ ± 0.3‰. Average $\delta^{13}C$ values were lowest during Period II, ranging from -18.4‰ to -21.1‰ with a mean of -19.5‰ ± 0.7‰, and were highest during Period I, ranging from -18.2‰ to -18.9‰ with a mean of -18.6‰ ± 0.5‰. Unfortunately, the extremely small sample sizes for both Periods III and I, as well as problems with measurement precision and the large standard deviation associated with the mean value for each time period, make it difficult to evaluate the significance of these temporal differences. Furthermore, the higher $\delta^{13}C$ values during Period I may be explained by a slight trophic level effect of breast-feeding, since the Period I individuals are both infants under six months of age. Given a trophic level effect for carbon in nursing infants of approximately +1‰, Period I $\delta^{13}C$ values for adults would be consistent with Period III and II data and thus there is no clear evidence for a change in $\delta^{13}C$ over time.

Average $\delta^{15}N$ values were lowest during Period III (8.1‰± 0.5‰) and highest during Period I (10.2‰ ± 1.6‰) (Figure 5.7). However, since the Period I individuals are both infants, the high nitrogen ratios are likely a trophic level effect of breast-feeding and, if the infant values are shifted on a trophic effect of approximately +2.5 to +3.0‰, the Period I values are more in line with Period III and II data . The standard deviation of the samples, as well as the precision of analysis, reveals overlap in nitrogen values between the three time periods, although a slight trend for increasing $\delta^{15}N$ over time is evident. However, the small and unequal sample sizes across the three time periods precludes any conclusive evaluation of change in nitrogen values, and hence dietary change, across the three time periods.

Environmental Reconstruction

Despite the enormous growth of northern Mesopotamian civilization during the third millennium, archaeological and historical records point to a period of marked environmental degradation between 2400 and 2200 B.C. (Courty, 1994; Issar, 1995; Jacobsen, 1982; Weiss, 1996; Weiss and Courty, 1993; Weiss et al., 1993). This deterioration apparently culminated about 2200 B.C. when, following a volcanic eruption, northern Mesopotamian cities such as Tell Leilan collapsed (e.g. Otterman and Starr, 1995; Weiss, 1996; Weiss et al., 1993). According to several researchers, a sudden shift in climate resulted in severe drought that inhibited agriculture and forced the inhabitants of the northern Mesopotamian centres to abandon their cities and move south (Weiss, 1996; Weiss and Courty, 1993; Weiss et al., 1993). It was not until

approximately 300 years later that civilization returned to the Habur Plains (Weiss and Courty, 1993; Weiss et al., 1993).

If environmental degradation and drought can be implicated in the collapse of northern Mesopotamia, the $\delta^{13}C$ and $\delta^{15}N$ values of third millennium skeletal samples from Tell Leilan should reveal isotopic signs of aridity, such as high nitrogen ratios (Ambrose, 1991, 1993). Additionally, if soils were still recovering from drought following the collapse, then $\delta^{13}C$ values from Period I might indicate the cultivation of more drought-tolerant C_4 plants. Compared with Period III nitrogen ratios, average $\delta^{15}N$ values from Period II samples were slightly higher (Figure 5.7), indicating a possible trend towards increased water-stress during the pre-collapse period. However, the difference between average nitrogen values for Period III and II is on the order of only 1‰, with significant overlap. The small number of Period III individuals confounds the assessment of any trend in $\delta^{15}N$ during the time period leading up to the abandonment of Tell Leilan. Additionally, the estimation of water-stress during Period I is impractical given the sample demographics: Period I is represented by infants alone, who may have elevated nitrogen ratios due to a trophic level effect of breast-feeding. Average carbon isotope ratios are highest during Period I, which may indicate the cultivation of more drought-adapted C_4 plants. However, these higher $\delta^{13}C$ values may also reflect a slight trophic level effect for breast-feeding infants.

The abrupt climatic change hypothesis generated by Weiss and his colleagues, however plausible, remains a subject of debate among archaeologists and Near Eastern specialists. Many researchers have been reluctant to blame the fall of Akkad solely on a sudden shift in climate. Political struggle, internal dissension, agricultural overintensification, and a failure to adapt to environmental change have been the preferred explanations for the collapse of northern Mesopotamian cities (e.g. Rosen, 1995, 1997; Wilkinson, 1994; Yoffee, 1995). The isotopic analysis of human remains from northern Mesopotamian sites has the potential to provide valuable information regarding the nature of the collapse at the end of the third millennium B.C. The overlap in $\delta^{13}C$ and $\delta^{15}N$ values for each time period suggest that there was no demonstrable aridification at Tell Leilan. Given the small Period III and I sample sizes, the uneven age distribution across time periods, and the imprecise age estimates for Period II individuals, however, the results from this study cannot be said to provide any conclusive evidence either for or against environmental shifts consistent with a period of intense aridification.

Conclusions

The results of stable carbon isotope analysis of human bone collagen from Tell Leilan coincide with the archaeobotanical evidence for a diet rich in C_3 plants, namely wheat and barley. Furthermore, the $\delta^{15}N$ values obtained in this study are in accord with a dietary model, derived from archaeozoological and botanical data, that includes the

consumption of nitrogen-rich meat (e.g. sheep, goats, and cattle) and nitrogen-depleted legumes (e.g. lentils). Additionally, the high nitrogen ratios in infants under six months of age, particularly in one individual, suggest a trophic level effect in this population due to breast-feeding.

The comparison of carbon and nitrogen isotope ratios over three time periods indicates that dietary adaptations at Tell Leilan did not change from 2900 to approximately 1900 B.C., even though soil analyses and geoclimatic data have revealed significant changes in land use conditions during the mid to late third millennium. Archaeological data demonstrate that, at approximately 2200 B.C., people fled en masse from cities across the northern Mesopotamian plains. Several researchers have attributed this large-scale abandonment to an abrupt climatic shift, perhaps brought on by an El Niño phenomenon (in Weiss, 1996) or the deposition of volcanic dust (Otterman and Starr, 1995). Seasonal rains apparently became insufficient to support agriculture at Tell Leilan and, as a result of increased winds, crops soon became blanketed in dust. If the climatic change hypothesis is true, the northern Mesopotamians would have lost a principal source of food and abandonment may have been the only option. The isotopic analysis of human remains from Tell Leilan does not provide any concrete evidence in support of an altered climate regime during the third millennium. The small sample size and the unequal distribution of ages across the three time periods investigated, however, renders this conclusion tentative.

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Table 5.1. Chronology and age/sex distribution of the skeletal sample

Burial Number	Leilan Period	Date (ca. B.C.)	Biological Age	Sex
L79 op1 108 B.1	IIIa	2900-2800	adult	unknown
L80 op1 61 Strat. 19	IIIc	2700-2600	adult	male
L80 op2 33/36 B.1	II	2400-2200	adult	female
L85 op4A 18 B.3	II	2400-2200	adult	male
L85 op4A 18 B.4	II	2400-2200	adult	unknown
*L89 76 E20 B.1 area 4**	II	2400-2200	infant	unknown
TL89 76 E20 B.2†	II	2400-2200	subadult	unknown
*L89 76 E20 B.3**	II	2400-2200	infant	unknown
L89 76 E20 B.6	II	2400-2200	adult	unknown
L89 76 F20 rm.6 B.7	II	2400-2200	adult	unknown
L89 76 F20 B.5/9	II	2400-2200	adult	unknown
TL87 77 G01 B.1	II	2300-2200	adult	unknown
L87 77 G01 B.2†	II	2300-2200	subadult	female
L87 77 G01 B.3†	II	2300-2200	subadult	male
L87 57 FG05 65 (346)‡	I	1900-1728	infant	unknown
L87 57 FG05 64 B.2‡	I	1900-1728	infant	unknown

* infant 6 months or older
† approximately 15 years of age
‡ infant under 6 months of age

Table 5.2. $\delta^{13}C$, $\delta^{15}N$ and C/N ratios for bone collagen, including replicate rib samples

Burial Number	Sample Number	Leilan Period	$\delta^{13}C$ (‰ PDB)	$\delta^{15}N$ (‰ AIR)	C/N
L79 op1 108 B.1	a	IIIa	-20.4	7.7	3.75
	b		-19.2	8.1	3.73
	c		-19.1	7.3	3.41
L80 op1 61 Strat.19	a	IIIc	-18.8	8.1	3.31
	b		-19.4	8.7	3.57
L80 op2 33/36 B.1	a	II	-19.8	8.8	3.69
	b		-20.0	8.7	3.67
L85 op4A 18 B.3	a	II	-19.6	8.9	3.41
	b		-20.5	9.3	3.56
	c		-19.4	9.3	3.86
	d		-19.2	8.3	3.32
L85 op4A 18 B.4	a	II	-19.2	9.0	3.64
	b		-19.1	9.1	3.74
*L89 76 E20 B.1 area 4**	a	II	-19.3	9.2	3.56
TL89 76 E20 B.2	a	II	-18.8	10.3	3.65
	b		-19.2	9.5	3.31
*L89 76 E20 B.3**	a	II	-20.3	7.6	3.24
L89 76 E20 B.6	a	II	-19.1	10.2	3.45
	b		-19.2	10.2	3.43
	c		-19.3	8.9	3.18
L89 76 F20 rm.6 B.7	a	II	-19.0	9.6	3.45
	b		-18.9	8.5	3.41
L89 76 F20 B.5/9	a	II	-19.9	9.5	3.46
	b		-19.6	9.2	3.43
	c		-19.3	8.4	3.32
TL87 77 G01 B.1	a	II	-21.1	8.7	3.88
L87 77 G01 B.2	a	II	-18.4	10.5	3.40
L87 77 G01 B.3	a	II	-19.0	8.1	3.37
L87 57 FG05 65 (346)‡	a	I	-18.9	9.1	3.26
L87 57 FG05 64 B.2‡	a	I	-18.2	12.0	3.60
	b		-18.1	10.7	3.20

* infant 6 months or older ‡ infant under 6 months of age

Table 5.3. Average $\delta^{13}C$ and $\delta^{15}N$ values of bone collagen for the 16 individuals from Tell Leilan

Burial Number	Leilan Period	n	$\delta^{13}C$ (‰ PDB)				$\delta^{15}N$ (‰ AIR)			
			Min.	Max.	Mean	S.D.	Min.	Max.	Mean	S.D.
L79 op1 108 B.1	IIIa	3	-19.1	-20.4	-19.5	0.7	7.3	8.1	7.7	0.4
L80 op1 61 Strat.19	IIIc	2	-18.8	-19.4	-19.1	0.4	8.1	8.7	8.4	0.4
L80 op2 33/36 B.1	II	2	-19.8	-20.0	-19.9	0.2	8.7	8.8	8.8	0.1
L85 op4A 18 B.3	II	4	-19.2	-20.5	-19.7	0.6	8.3	9.3	9.0	0.5
L85 op4A 18 B.4	II	2	-19.1	-19.2	-19.2	0.1	9.0	9.1	9.0	0.1
*L89 76 E20 B.1 area 4**	II	1	-19.3	-19.3	-19.3	-----	9.2	9.2	9.2	-----
TL89 76 E20 B.2	II	2	-18.8	-19.2	-19.0	0.3	9.5	10.3	9.9	0.6
*L89 76 E20 B.3**	II	1	-20.3	-20.3	-20.3	-----	7.6	7.6	7.6	-----
L89 76 E20 B.6	II	3	-19.1	-19.3	-19.2	0.1	8.9	10.2	9.8	0.7
L89 76 F20 rm.6 B.7	II	2	-18.9	-19.0	-19.0	0.1	8.5	9.6	9.1	0.7
L89 76 F20 B.5/9	II	3	-19.3	-19.9	-19.6	0.3	8.4	9.5	9.0	0.6
TL87 77 G01 B.1	II	1	-21.1	-21.1	-21.1	-----	8.7	8.7	8.7	-----
L87 77 G01 B.2	II	1	-18.4	-18.4	-18.4	-----	10.5	10.5	10.5	-----
L87 77 G01 B.3	II	1	-19.0	-19.0	-19.0	-----	8.1	8.1	8.1	-----
L87 57 FG05 65 (346)‡	I	1	-18.9	-18.9	-18.9	-----	9.1	9.1	9.1	-----
L87 57 FG05 64 B.2‡	I	2	-18.1	-18.2	-18.2	0.1	10.7	12.0	11.3	0.9

* infant 6 months or older

‡ infant under 6 months of age

Table 5.4. Carbon and nitrogen stable isotope statistics by time period

Leilan Period	n	$\delta^{13}C$ (‰ PDB)				$\delta^{15}N$ (‰ AIR)			
		Min.	Max.	Mean	S.D.	Min.	Max.	Mean	S.D.
III	2	-19.1	-19.5	-19.3	0.3	7.71	8.38	8.05	0 5
II	12	-18.4	-21.1	-19.5	0.7	7.57	9.89	9.04	0 8
I	2‡	-18.2	-18.9	-18.6	0.5	9.11	11.33	10.22	1 6

‡ infants under 6 months of age

Figure 5.1 - Map of ancient Mesopotamia showing the location of Tell Leilan (adapted from Gibbons, 1993)

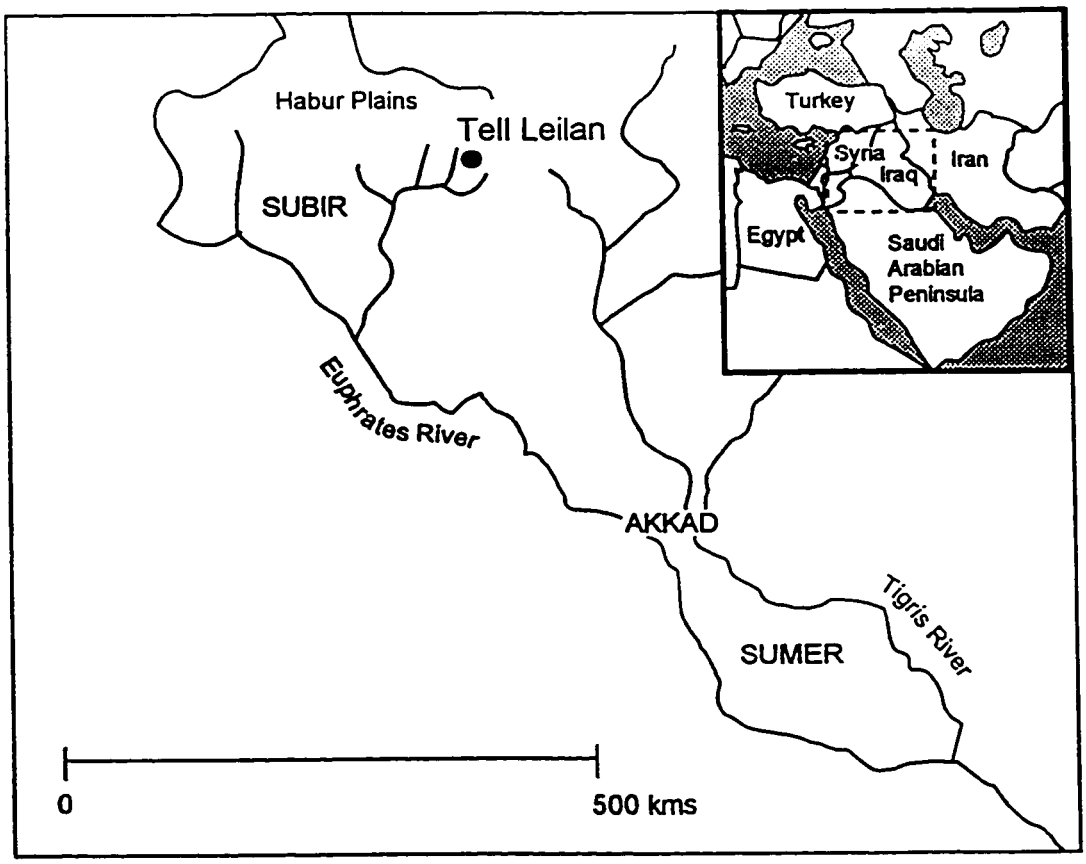


Figure 5.2. Variation in $\delta^{13}C$ over four age groups

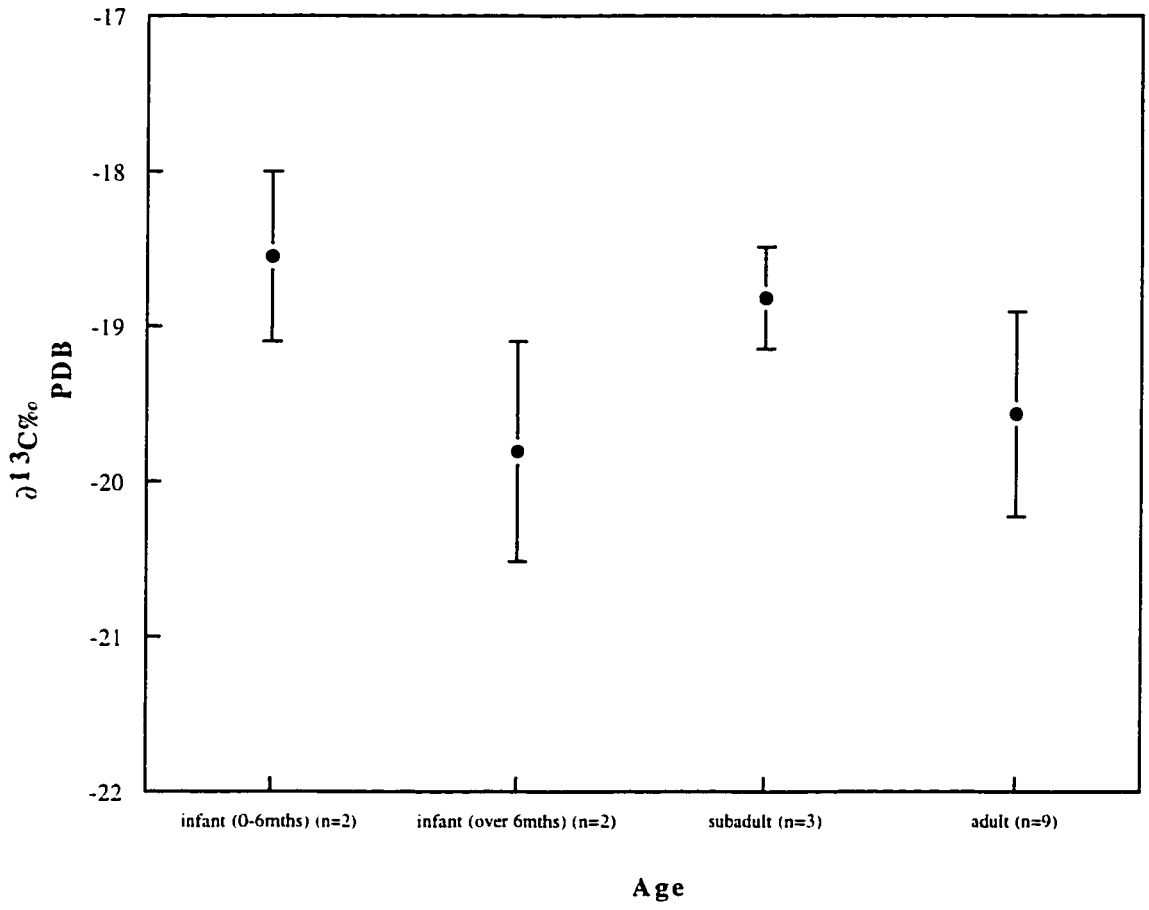


Figure 5.3. Variation in $\delta^{15}N$ over four age groups

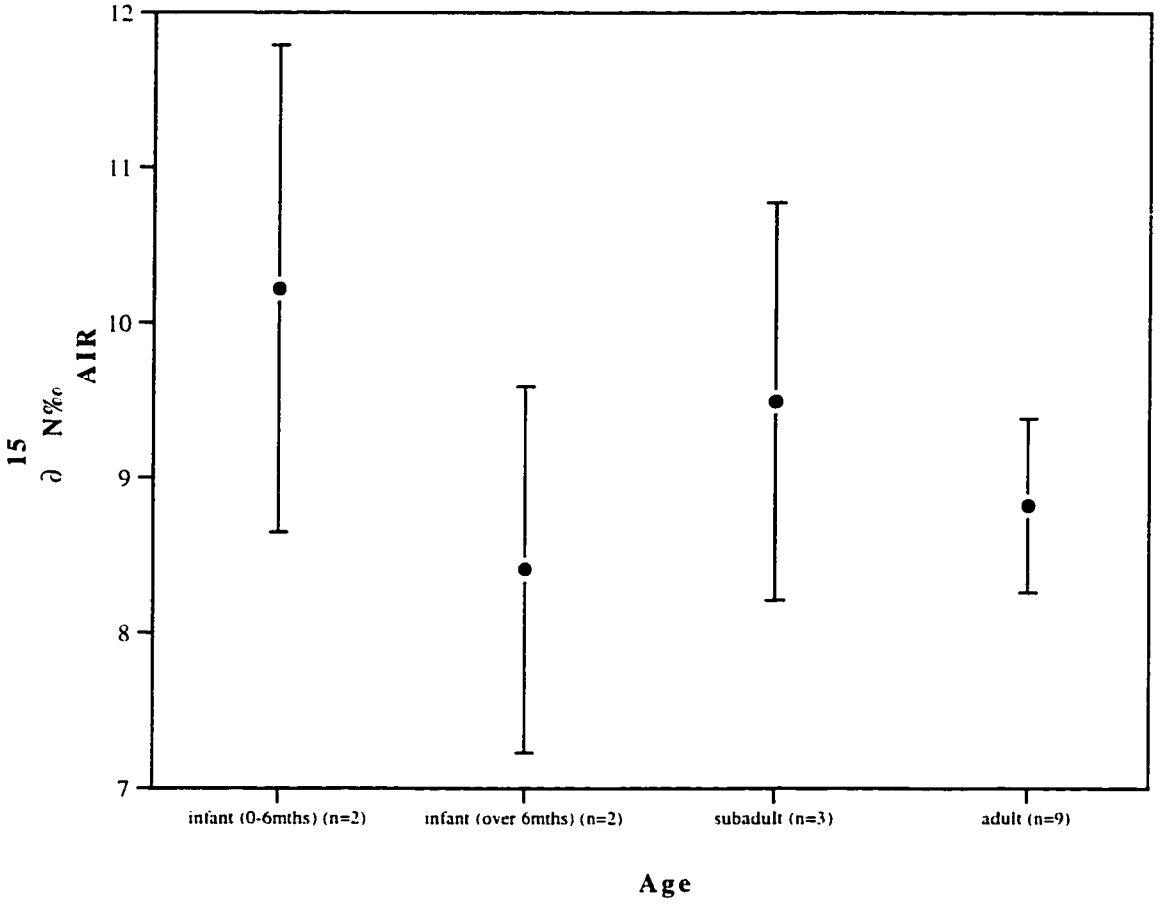


Figure 5.4. Distribution of stable carbon and nitrogen isotopes in marine and terrestrial systems (modified from Ambrose, 1993)

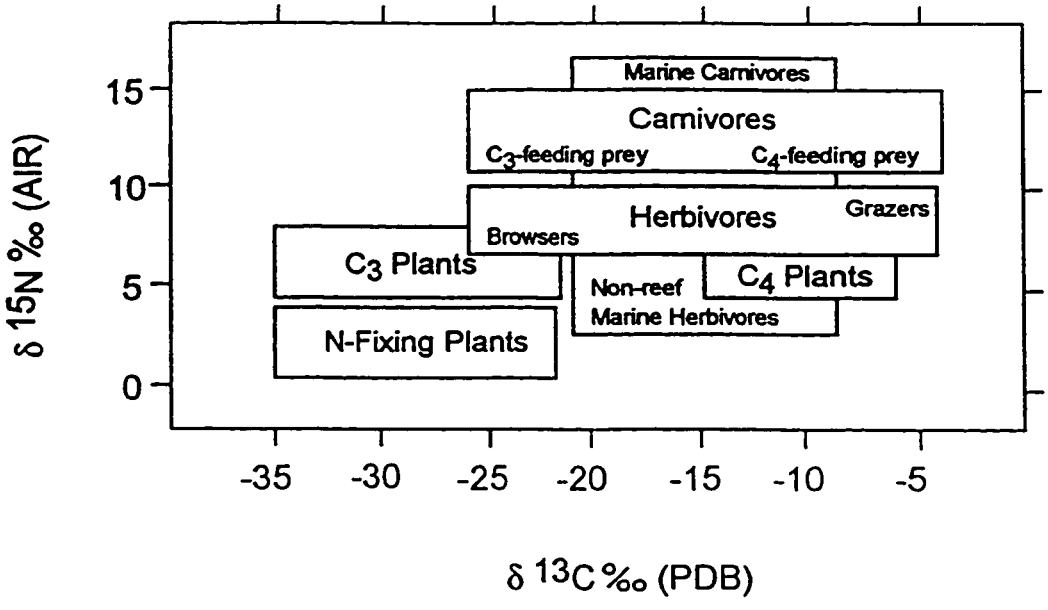


Figure 5.5. Average $\delta^{13}C$ and $\delta^{15}N$ values of bone collagen from Tell Leilan human rib samples
· (data with no error bars represent single measurements)

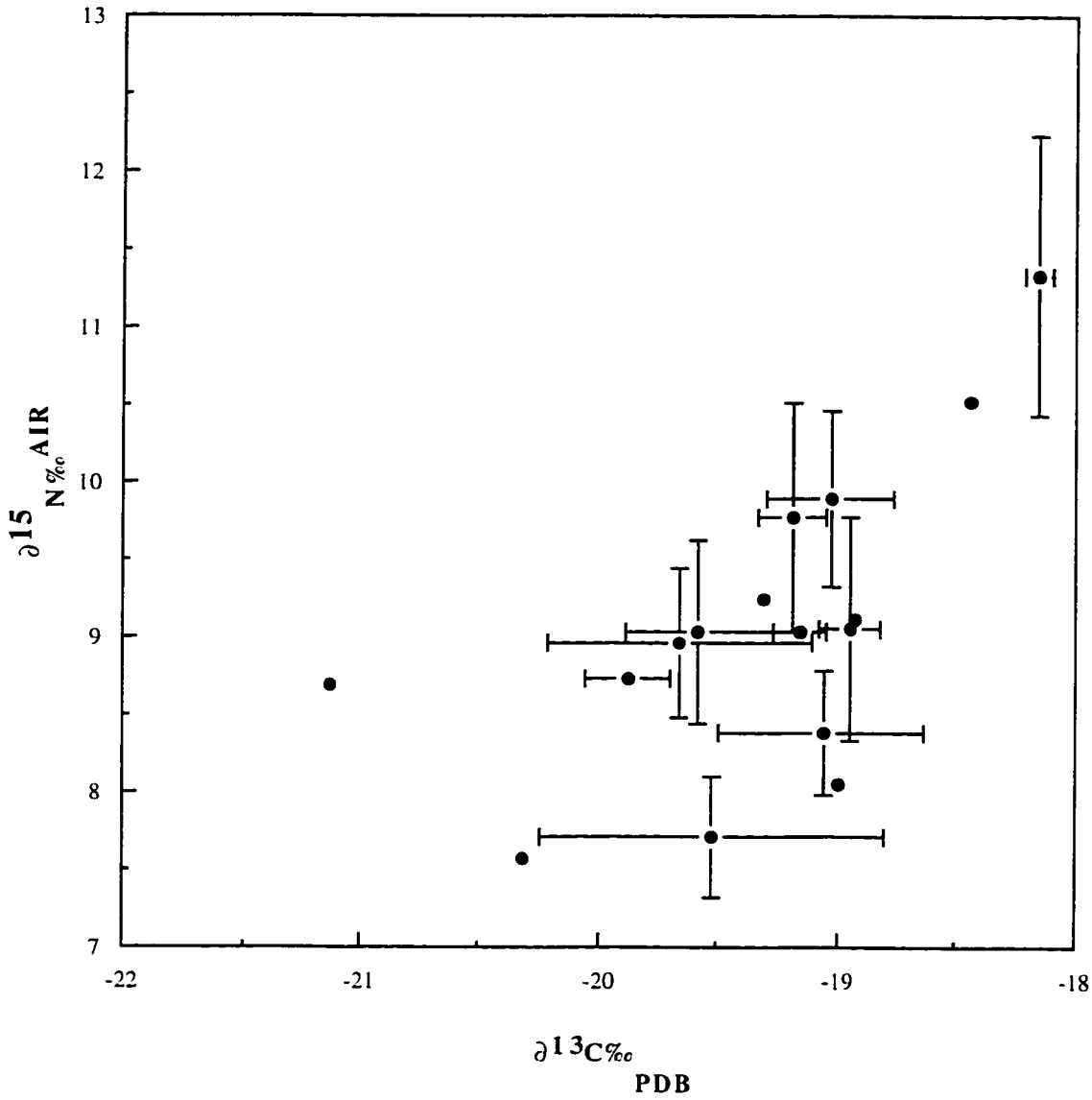


Figure 5.6. Temporal variation in $\delta^{13}C$ at Tell Leilan

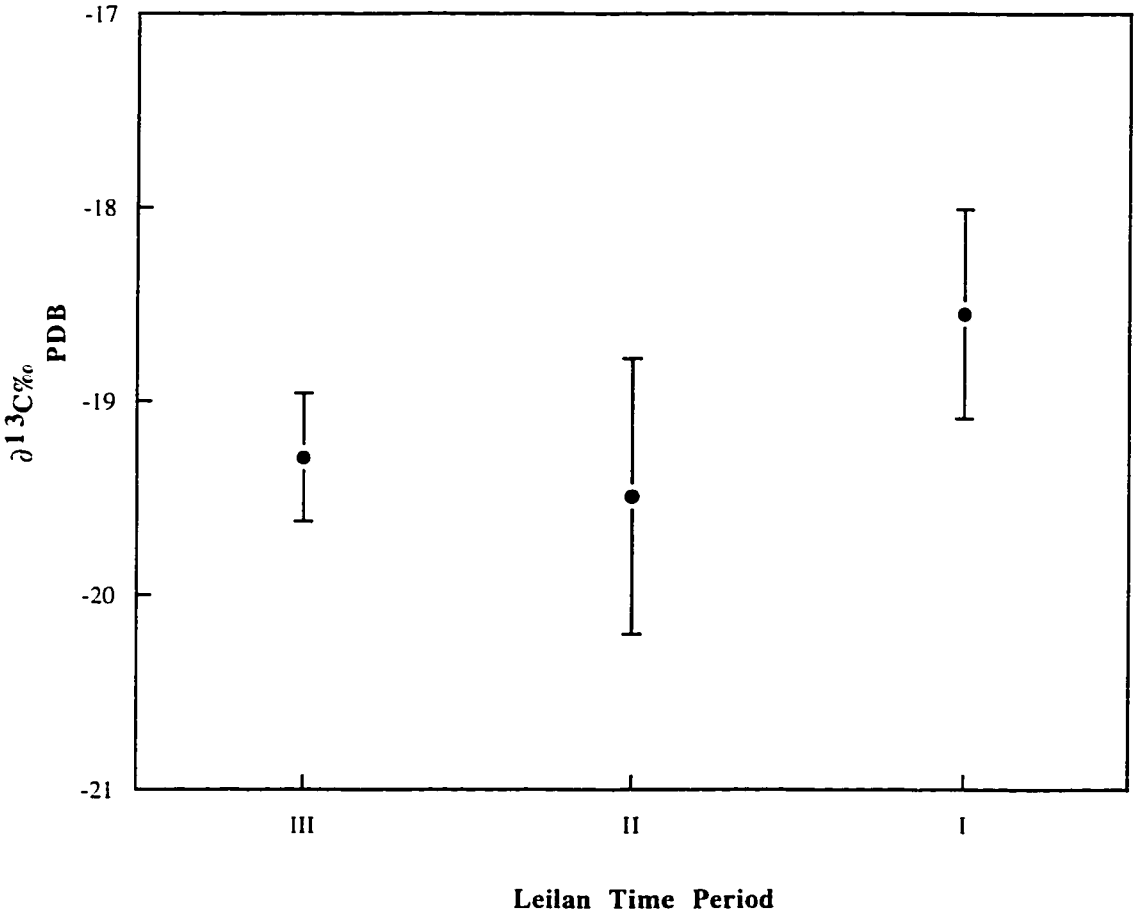
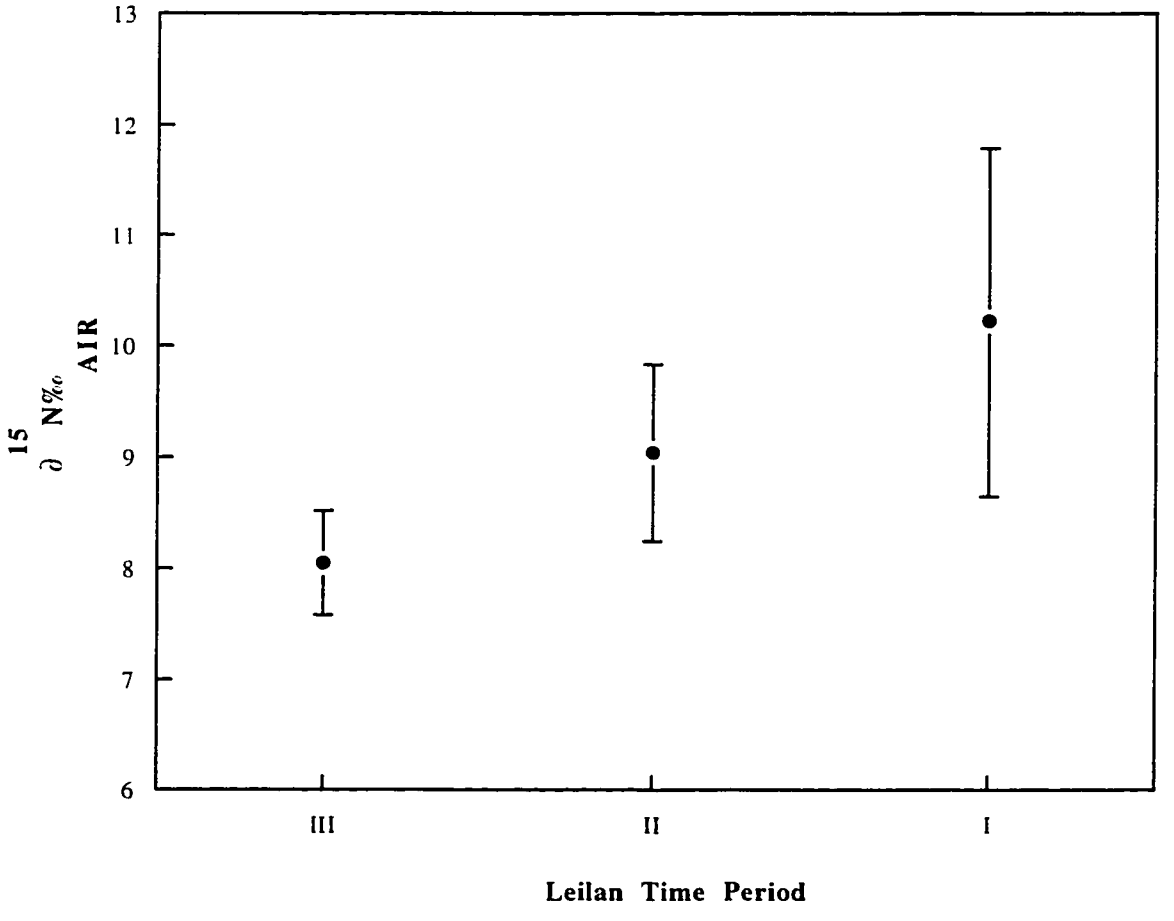


Figure 5.7. Temporal variation in $\delta^{15}N$ at Tell Leilan



Chapter 6

Conclusions

The field of stable isotopy for dietary reconstruction is growing rapidly, as researchers strive to refine sample preparation methods and define methodological and interpretive areas of uncertainty. As such, the practical importance of stable isotope analysis has come to the forefront of archaeology and researchers are increasingly relying on stable isotopes to reconstruct the lifeways of past peoples. Palaeoanthropological applications of stable isotopes have broadened to include the estimation of prehistoric environmental conditions, the detection of disease in ancient populations, and the determination of weaning age in antiquity. Substantial insights into prehistoric dietary and land use conditions using the stable isotopes of carbon and nitrogen have been achieved and the promise of stable isotopes as reliable and accurate indicators of palaeodietary adaptations has heightened anthropological interest in this technique.

In this study, rib samples from 16 different individuals from the third millennium site of Tell Leilan in northeastern Syria were analyzed for the stable isotopes of carbon and nitrogen. $\delta^{13}C$ values suggest a terrestrial-based diet rich in C_3 plants and nitrogen isotope ratios indicate the consumption of meat and legumes. Archaeobotanical and archaeozoological analyses are consistent with these findings: botanical remains indicate an abundance of wheat, barley, and lentils at the site and faunal analyses suggest that goat, sheep, and cattle were likely farmed at Tell Leilan. High $\delta^{15}N$ values in infants up to six months of age also suggest a potential trophic level effect of breast-feeding in this population. Despite recent archaeological and geoclimatic research, which has pointed to a period of intense aridification during the mid-third millennium, the chronological comparison of carbon and nitrogen isotope ratios over three time periods indicates that dietary adaptations at Tell Leilan did not change between 2900 and approximately 1900 B.C. Moreover, the $\delta^{13}C$ and $\delta^{15}N$ values do not show any isotopic signs of aridity, such as elevated nitrogen ratios. However, given the small sample size and inconsistent age distribution across time periods, these interpretations should be considered tentative.

The reconstruction of ancient human dietary patterns using stable isotopes is predicated on the assumption that the isotopic signals in archaeological bone reflect the isotopic composition of the living individual. Accordingly, the contamination and post-mortem alteration of isotopic ratios in bone collagen, as well as the isolation of collagen residues with biological integrity, are problems that must be considered. As a first check for collagen integrity, the method of demineralization must take into account the preservation and age of skeletal samples. Although acid concentration had no appreciable effect on $\delta^{13}C$ and $\delta^{15}N$ values in this study, the demineralization process was most satisfactory when 1% HCl was used. Stronger acid solutions resulted in "flaky" collagen pseudomorphs that did not conform with the standards of sample acceptance. The utility of removing humic

contaminants with sodium hydroxide was also examined and, in this study, the NaOH completely dissolved the initially prepared set of collagen pseudomorphs. Since NaOH treatments can result in a loss of collagen, and since the yields from later samples taken from the same individuals suggested poor collagen preservation, it was concluded that low collagen yield was a factor in the NaOH destruction of the collagen residues. These findings are relevant to isotopic studies of aged bone samples where collagen preservation is questionable.

The results from this study have implications for the isotopic analysis of Near Eastern skeletal material. The importance of proper bone demineralization and the ineffectiveness of treating poorly preserved samples with NaOH have been aptly demonstrated.
Dietary interpretations based on $\delta^{13}C$ and $\delta^{15}N$ values support archaeobotanical and archaeozoological evidence from Tell Leilan. Chronological comparisons of carbon and nitrogen isotope ratios, however, do not support a model of an altered climate regime during the mid-third millennium B.C. in northern Mesopotamia. As archaeological investigations at Tell Leilan continue, the isotopic analysis of plant and faunal remains will be necessary in order to adequately interpret dietary adaptations and environmental conditions.

IMAGE EVALUATION TEST TARGET (QA-3)

150mm

6"

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