STABLE ISOTOPE ANALYSIS OF FAUNA AND SOILS FROM SITES IN THE EASTERN FREE STATE AND WESTERN LESOTHO, SOUTHERN AFRICA: A PALAEOENVIRONMENTAL INTERPRETATION

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A thesis submitted to the Faculty of Science, University of Cape Town, in fulfilment of the requirements for the degree of Master of Science, Department of Archaeology.

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DECLARATION

I declare that this thesis is my own unaided work unless otherwise acknowledged. It is submitted for the degree of Master of Science in the University of Cape Town. It has not been submitted before for any other degree or examination in any other university.

- Signed by candidate

14TH day of April, 1997

ABSTRACT

This thesis examines the use of stable carbon isotopes as a means of reconstructing the palaeoenvironment of the Caledon River Valley of the eastern Free State, South Africa, and western Lesotho. In doing so, this work draws upon previous studies that have shown that the distinct distribution and $\delta^{13}C$ values of C_3 and C_4 grasses are influenced by seasonality of rainfall and growth season temperatures. In general, C_3 grasses dominate in areas where conditions are cool/moist during the growth season, while C_4 grasses characterize those that are warm/arid. The isotopic composition of the grasses of an area, and thus climatic and environmental data, is passed along the trophic levels, through dietary intake by grazers, and decomposition into soil sediments.

By measuring the $^{13}C/^{12}C$ ratios of carbon extracted from the calcified tissues of grazers and soil organic matter recovered from within an archaeological context, a palaeoenvironmental sequence has been reconstructed for the study area for the last 13 500 years. Results have shown that although C_4 grasses have dominated, the presence of C_3 grasses, at various times during this period, suggest that growth season temperatures fluctuated temporally and spatially.

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Soil, climate, vegetation and fauna are no mere background to human cultures, but they are the very seed-bed in which they grow and which in turn they helped to form.

(Grahame Clark 1960:175, 176)

CHAPTER 1

INTRODUCTION

The aim of this study is to reconstruct the terminal Pleistocene and Holocene palaeoenvironment of the Caledon River Valley through the use of stable carbon isotopic analysis of ungulate herbivores and soil organic matter. A study of this nature was deemed necessary for two reasons. First, there is a need to develop a well dated palaeoenvironmental database for this area of the Grassland Biome in order to contribute to the understanding of climatic fluctuation in the interior of southern Africa during this time period. It was decided to use archaeologically derived materials as the main source of data, as this would provide a means of monitoring environmental change through the analysis of dated deposits. Secondly, knowledge of these past environments provides a necessary contribution towards archaeological work carried out in the area, as the results have direct association with cultural assemblages. This project thus forms a facet of two ongoing archaeological investigations in the eastern Free State (L. Wadley) and Lesotho (P. Mitchell).

Analysis of the ratio of $^{13}C/^{12}C$ in faunal material and soil sediments, as derived from the isotopic composition of vegetation, forms the basis of this palaeo-environmental reconstruction. The natural distribution

of vegetation types, with distinct $^{13}C/^{12}C$ ratios, is influenced by seasonality of rainfall and temperature. In general, grasses utilizing the C_4 photosynthetic pathway are characteristic of a warm growth season, whereas those with a C_3 pathway tend to reflect cool/moist conditions during the growth season. The relative portion of C_3/C_4 grasses present in the past will be reflected in the calcified tissues of grazers, as well as in soil organic matter.

It was decided, for the purposes of this study, to use as broad a database as possible. For this reason archaeological materials from five different sites have been sampled for analysis. Teeth and bone from ungulate herbivores from the sites of Rose Cottage Cave and Twyfelpoort in the eastern Free State, and the western Lesotho sites of Tloutle, Ha Makotoko, and Ntloana Tsoana are examined (Fig. 1.1). Further isotopic analysis is undertaken on soil organic matter from soils and sediments obtained from Rose Cottage Cave and its vicinity.

The fact that these independent lines of evidence overlap in time enables the comparison of one data set with another. Furthermore, the use of two different isotopic sources, soils and ungulate skeletal remains, provides insight into both the vegetation directly associated with the sites (soils), and the more regional

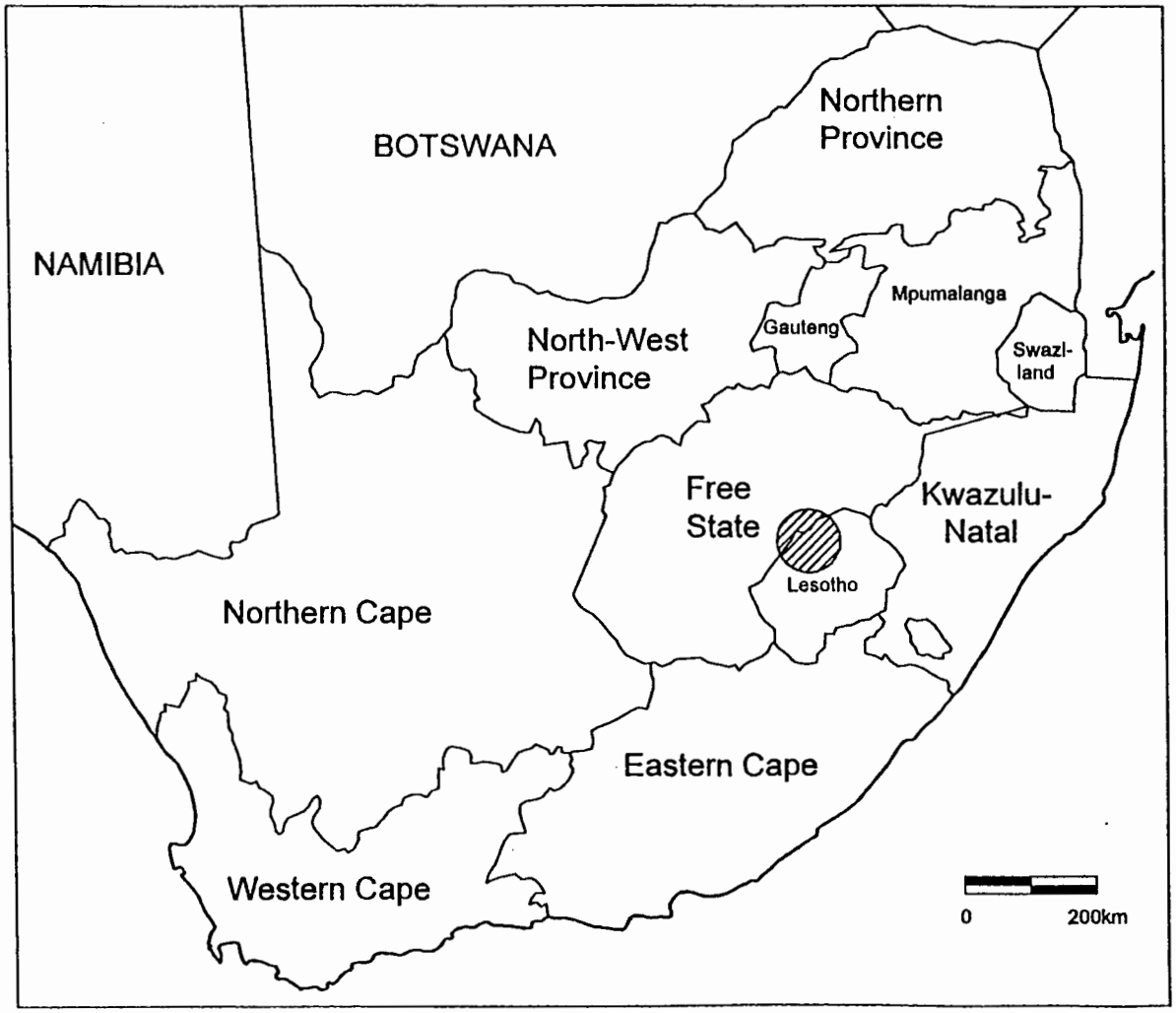

Figure 1.1 Location of study area in relation to southern Africa

environment of the Grassland Biome (migratory ungulates). Conceptually, this multifaceted analysis stems from the stable isotopic studies carried out on animals, plants, and soils from East Africa to reconstruct patterns of environmental change during the Holocene (Ambrose and DeNiro 1986, 1989; Ambrose and Sikes 1991; Cerling *et al*. 1991).

Chapters two and three delineate the methods and methodology behind the isotopic analyses used in this study. The methodology draws strongly on the pioneering work of Vogel *et al*. (1978) and Vogel (1978), which demonstrated that grasses growing in the winter and summer rainfall areas of southern Africa display distinctly different stable carbon isotopic values from one another. Furthermore, their studies showed that these different values are passed along the trophic levels and subsequently reflected in the calcified tissues of archaeologically recovered grazers. Methods of extracting collagen and biological apatite from calcified tissues are based on the successful processes outlined by Sealy (1986) and Lee-Thorp (1989). This study focuses predominantly on the use of enamel apatite, as collagen was not always preserved in the archaeological samples. However, when collagen is available it was deemed necessary to carry out the analyses of both collagen and apatite. Previous studies (Lee-Thorp *et al*. 1989) showed that the isotopic

difference between collagen and apatite provides an important means of assessing the reliability of apatite values.

These chapters also deal with the isotopic analysis of soil organic matter. Studies of this nature have shown that soil organic matter will have an isotopic value reflecting that of the vegetation in place at the time of soil formation (for examples see O'Brien and Stout 1978; Dzurec *et al.* 1985). Methods for extracting carbon from the soils organic matter follow those outlined by Bond *et al.* (1994) and Cerling *et al.* (1991).

Chapter four discusses the modern vegetation of the study area situated within the Grassland Biome. Factors, such as climate, soils, altitude, and humans, which determine and affect the development of the Grassland Biome are examined. An understanding of these modern parameters is necessary to provide a starting point from which to identify possible underlying causes of change in the past. Historically documented migratory routes of ungulate herbivores are also given consideration. It is necessary to establish the nature and extent of these routes as these animals appear to have roamed through a number of different micro-environments within the Grassland Biome that display isotopic variability.

The archaeological sites and surrounding environments are introduced in Chapter five, and the dates and context of the faunal and soil samples are presented. The archaeological context of each specimen is important as it has a bearing on the integrity of the sample in relation to other palaeoenvironmental evidence and cultural remains.

The results of this study are presented and discussed in Chapter six. This chapter is divided into three parts. The first part is designed to establish a modern stable carbon isotopic baseline for the study area. This provides a cornerstone from which to understand the extent of vegetational change in the past. Secondly, the isotopic results from the study of fauna dated to between the terminal Pleistocene and middle Holocene are presented and interpreted. These findings are discussed in the light of other palaeoenvironmental interpretations from these and other sites in the more general region of the Grassland Biome and adjacent biomes. The primary concern of this research is to reconstruct a local environmental history to go with that of the archaeology of the Caledon River Valley. Hence, the focus of this regional comparison is therefore mainly on data from sites in the eastern Free State and Lesotho. Lastly, isotopic results from fauna and soil organic matter dated to within the last 2300 years are discussed. Interpretation of the data from

this period proved difficult as the availability of material was fairly limited. Nevertheless, a tentative climatic scenario is provided, as well as an assessment of human impact on the environment.

Chapter seven concludes the thesis with a synopsis of the study, and provides some recommendations for future isotopic research in the study area.

CHAPTER 2

STABLE CARBON ISOTOPES IN THE ENVIRONMENT

The use of stable carbon isotopes as a means of reconstructing past environments requires an understanding of how carbon is distributed within an ecosystem, and for interpretation to take place two fundamental criteria must be observed. First, vegetational sources of carbon forming the base of the food web, must be isotopically distinct. Secondly, the isotopic signature in various parts of the systems must be preserved or should change in a predictable manner. This chapter examines these requirements in relation to variability of stable carbon isotopes in terrestrial plants and the transferral of isotopic signatures to terrestrial animals and soil sediments.

Stable Isotope Background

Isotopes are forms of the same element that contain the same number of protons and electrons, but differ in the number of neutrons. Hence, they will have the same chemical properties, but vary in atomic mass. Isotopes are referred to in relative terms: "stable" isotopes do not under go radioactive decay at a measurable rate, whereas "unstable" or radioactive ones do (Hoefs 1987: 1). Many naturally occurring elements have two or

more stable isotopes; one isotope of the element is usually present in far greater abundance than the other(s). Elemental carbon ^{12}C, for example, is approximately 100 times more abundant than ^{13}C.

Although the stable isotopes of an element are chemically similar and are able to participate in the same chemical or physical reaction, they react at different rates due to mass differences (Hoefs 1987:3,4). As a result, during a chemical, physical, or biological reaction the proportion of one isotope to another may change, causing a difference in the isotopic ratio between the starting and end products. This change is known as fractionation and forms the basis for tracing geological and biological variations in the environment.

Fractionation

Fractionation in most geological and biological systems is the result of either kinetic or equilibrium isotope effects (Craig 1953, 1954; Vogel 1980; Hayes 1982; Hoefs 1987:11,12; O'Leary 1988). Kinetic isotope effects occurs "...when the rate of a chemical reaction is sensitive to the atomic mass at a particular position in one of the reacting species" (Hayes 1983:3), resulting in differing reaction rates for the various isotopes. Equilibrium isotope effects ensure that the heavier

isotope accumulates in the more stable part of a system, as molecules containing heavier isotopes react or diffuse more slowly and form stronger bonds than the lighter isotope (Griffiths 1991:257).

Fractionation is measured in terms of the relative isotope abundance of a sample compared to that of a known standard, as the absolute abundance is difficult to determine (Hayes 1983:6). The relative isotope abundance is determined by mass spectrometry and is expressed in delta (δ) notation measured in parts per mil (‰). Values are based on the formula:

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

where δ is expressed in terms of the heavier isotope, for example enriched or depleted in ^{13}C, and R is the abundance ratio of heavy to light isotope, in this case $^{13}C/^{12}C$.

In kinetic reactions the fraction that takes place between the isotopic source and the product is expressed as:

$$\Delta = (\delta_{source} - \delta_{product}) / (1 + \delta_{product})$$

or

$$\Delta = \delta_{source} - \delta_{product}$$

For carbon, the source is atmospheric CO_2 and the product may be, for example, a plant.

Stable Carbon Isotope Sources

Although there exist freshwater and marine sources of carbon (see Boutton 1991 for review of isotopic variability), the focus of this study is on carbon in the terrestrial ecosystem; primarily tracing changes in the ratio of two naturally occurring stable isotopes of carbon, ^{13}C and ^{12}C, in plants, animals, and soil sediments (Fig. 2.1) In general, carbon enters the terrestrial ecosystem through atmospheric CO_2, which is taken up by green plants during photosynthesis to produce glucose. The glucose is in turn used to form carbohydrates and proteins that can be incorporated in the tissues of higher order consumers. Through respiration of plant and animals, and decomposition of these organisms into carbon-containing compounds, CO_2 is re-integrated into the ecosystem. The process of photosynthesis is expressed in the following equation:

Photosynthesis With Light Energy

$$\underset{\text{Water}}{6H_2O} + \underset{\text{Carbon Dioxide}}{6CO_2} + \text{Energy} \rightarrow \underset{\text{Glucose}}{C_6H_{12}O_6} + \underset{\text{Oxygen}}{6O_2}$$

Recent increase of CO_2 in the atmosphere from the burning

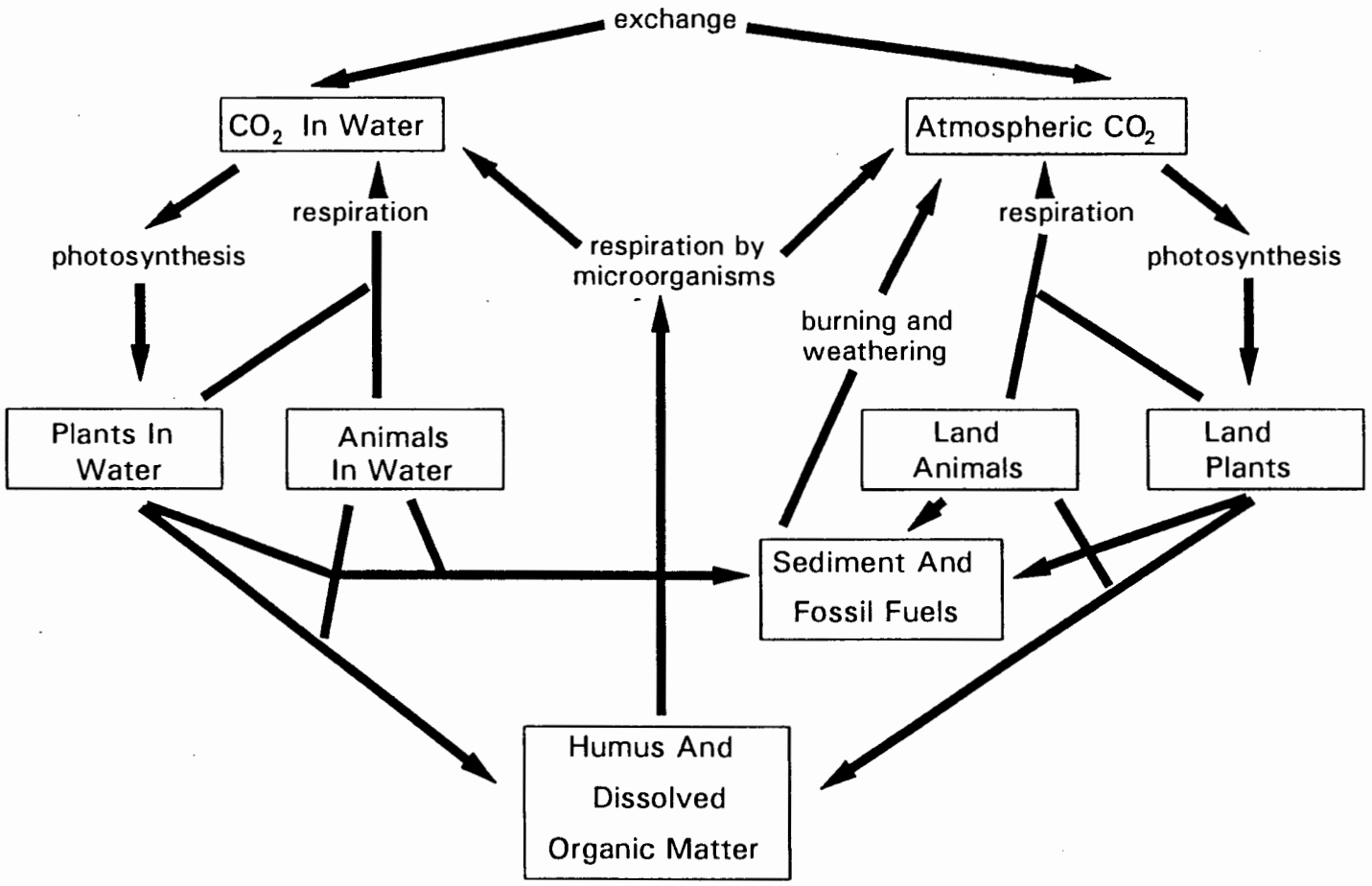

Figure 2.1 Schematic diagram of the carbon cycle (adapted from Alberts *et al.* 1983)

of fossil fuels has caused a depletion of ^{13}C in modern plants relative to preindustrial counterparts. Present atmospheric CO_2 has a $\delta^{13}C$ value of about -8‰. However, short and long term trend studies carried out on Antarctic ice-cores (Friedli *et al.* 1986), tree-rings (Freyer 1986), and modern and prehistoric plants (Marino *et al.* 1992) indicate that $^{13}CO_2$ has become 1 to 2‰ less abundant through time. In a study of spatial differences in $\delta^{13}CO_2$ values, it was found that the northern hemisphere is depleted approximately 0.2‰ in relation to the southern hemisphere (Keeling *et al.* 1978). This is attributed to greater amounts of anthropogenic CO_2 in the northern hemisphere, and relatively slow rates of mixing in the atmosphere.

Overall, the depletion in atmospheric $\delta^{13}CO_2$ value has been 1 to 2‰ since the beginning of the industrial period. If archaeologists are to use modern plants, animals, and soils as a baseline by which to measure and interpret the stable carbon isotopic composition of archaeological specimens, it is important to be aware of temporal and spatial changes in $\delta^{13}CO_2$ values.

Stable Carbon Isotopes In Terrestrial Vegetation

All terrestrial plants are depleted in ^{13}C in relation atmospheric CO_2, but abundance varies depending on which of the three photosynthetic pathways is utilized, C_3, C_4,

or CAM (Wickman 1952; Craig 1953, 1954; Calvin and Benson 1948; Kortschak *et al*. 1965; Hatch *et al*. 1967; Bender 1968; Smith and Epstein 1971; O'Leary 1988). Fractionation during photosynthesis causes a depletion of about -5‰ in C_4 and -19‰ in C_3 in plants, relative to atmospheric $\delta^{13}CO_2$. As a result C_3 and C_4 plants have distinct and non-overlapping $\delta^{13}C$ values. CAM plants are trimodal, having values that are either similar to C_3 or C_4 plants, or intermediate between the two.

Most plants use the Calvin-Benson photosynthetic pathway. This is commonly referred to as the C_3 process, since CO_2 is reduced to phosphoglyceric acid, a molecule containing three carbon atoms, as its first intermediate product (Calvin and Benson 1948). In this process, photosynthesis takes place via the enzyme ribulose diphosphate carboxylase (RuDP), a phosphorylated sugar. The enzyme RuDP discriminates strongly against $^{13}CO_2$ resulting in relatively low $\delta^{13}C$ values for C_3 plants. Values for these plants range from -34 to -23‰, with a mean of -26‰, and include most trees and shrubs, and grasses adapted to temperate or shaded forest conditions (Calvin and Benson 1948; Hatch *et al*. 1967; Smith and Epstein 1971). C_3 grasses are restricted primarily to temperate winter rainfall zones, but will occur at cool high altitudes in summer rainfall areas (Vogel *et al*. 1978; Tieszen *et al*. 1979; Livingston and Clayton 1980; Tieszen and Boutton 1989). As seen in Fig. 2.2, in

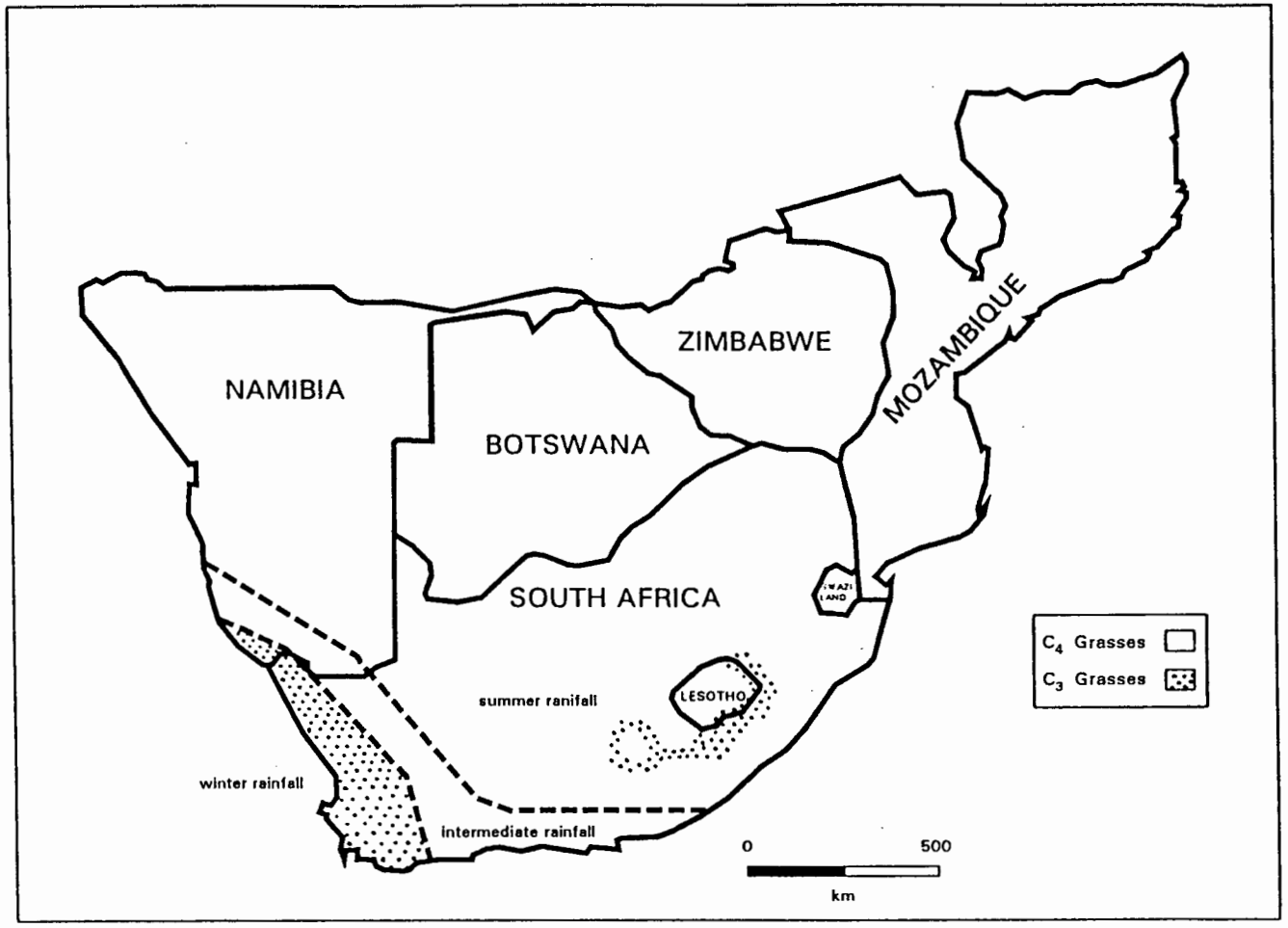

Figure 2.2 Approximate Distribution of C_3 and C_4 grasses in southern Africa (adapted from Vogel 1978)

southern Africa C_3 grasses dominate only in the winter rainfall areas of the Western Cape, and in the high-altitude Drakensberg and Maluti Mountains (Killick 1978; Vogel *et al*. 1978:212; Cowling 1983; Tainton 1984; Acocks 1988). In mountainous regions slope direction effects the altitudinal distribution of vegetation types, with C_3 plants growing at lower altitudes on south facing slopes than on sunny north facing ones (Cowling 1983).

The range of $\delta^{13}C$ values for C_3 plants is greater than that of C_4, reflecting in part the diverse environmental distribution of C_3 species. For example, in dense forest or closed-canopy conditions, the increase in both moisture and respired CO_2, and the reduction in light intensity result in $\delta^{13}C$ values that are more depleted than those of C_3 vegetation in open biomes (Medina and Minchin 1980; Ehleringer *et al*. 1987; van der Merwe and Medina 1991).

In the C_4 or Hatch-Slack pathway, CO_2 initially reacts with the enzyme phospho-enol-pyruvate carboxylase (PEP) to form malic acid and aspartic acid, each having four carbon molecules (Kortschak *et al*. 1965; Hatch *et al*. 1967). The CO_2 fixed in these acids is broken down by enzymes and then released into the Calvin-Benson pathway. The $\delta^{13}C$ values of C_4 plants are relatively high compared to C_3 plants, because the PEP enzyme does not

discriminate against $^{13}CO_2$ to the same degree as the RuDP enzyme (Ehleringer and Osmond 1991:290). For plants with the C_4 pathway $\delta^{13}C$ values range from -9 to -17‰, with a mean of -12‰ per mil (Smith and Epstein 1971). These plants are found in tropical and subtropical climates, where temperatures and light intensity are high during the growth season. C_4 species are mainly grasses adapted to warm and/or xeric environments, and cereal crops such as maize and millet (Bender 1968; Smith and Epstein 1971). In South Africa C_4 grasses are found predominantly in the summer rainfall areas of the interior and the eastern parts of the country, and throughout the lower altitudes of Lesotho (see Fig. 2.2) (Vogel *et al.* 1978).

The Crassulacean Acid Metabolism (CAM) pathway is used primarily by succulents, such as cacti and certain *Euphorbiaceae*. In CAM plants both RuDP and PEP are present and have $\delta^{13}C$ values that overlap with those of C_3 and C_4 plants, ranging from -10 to -22‰ (Bender *et al.* 1973; Osmond *et al.* 1973). There are two types of CAM plants: Obligated-CAM species, and Facultative- CAM species that will switch between the two photosynthetic pathways depending on environmental conditions (O'Leary 1988; Berry 1989:91; Ehleringer and Osmond 1991:291). On the whole, $\delta^{13}C$ values differentiate CAM plants from C_3 plants; distinction from C_4 plants can be made based on physiology (O'Leary 1988). The distribution of CAM

plants in Lesotho has not been extensively studied, but in South Africa they are common on the west coast of the Western Cape and in the Little Karoo.

Environmental Influences On The Distribution Of C_3 And C_4 Vegetation

The need for the various photosynthetic pathways is a response to temperature, light intensity, and the availability of water, factors which affect CO_2 concentrations (Smith 1980:21). At very low CO_2 concentrations, which exist when temperatures and light intensity are high and water is limited, C_4 plants carry out photosynthesis more efficiently than C_3 plants. Thus, C_4 plants have a higher optimum leaf temperature for photosynthesis, between 30 to 45°C, than C_3 plants, 10 to 25°C. To minimize water evaporation when opening the stomata, C_4 plants fix CO_2 at both a faster rate and at lower levels than C_3 plants, thereby, maximizing conversion of CO_2. As a result, C_4 plants are able to attain the same rate of photosynthesis as C_3 plants with less water loss (Tieszen *et al*. 1979:338; Campbell 1987).

A correlation exists between the type of vegetation cover and associated environmental conditions, with temperature and precipitation frequently reported as primary factors governing the proportion of C_3/C_4

vegetation in a particular area. Studies in North America have shown that along a temperature and precipitation gradient in grassland communities, C_3/C_4 plant biomass is linearly related to mean annual temperature and precipitation (Teeri and Stowe 1976; Boutton *et al.* 1980), with C_4 grasses found in hot/arid regions. In South Africa, Vogel *et al.* (1978:214) propose temperature as the most important factor controlling plant type distribution. Their studies found that during the growth/rainy season C_4 plants require a mean daily maximum temperature of at least 25°C and a minimum/nightly temperature above 8°C. Below this C_3 grasses become more successful. In South Africa, there is no precise relationship between temperature and precipitation. C_4 species occur in a mosaic of environments, such as arid desert, temperate high altitude grasslands, and the humid subtropical east coast (Vogel *et al.* 1978:214). It has been suggested that even in regions of high precipitation, plants have to cope with frequent dry spells and high rates of evaporation (Vogel *et al.* 1978:214)

The level of atmospheric CO_2 may also influence the distribution of C_3 and C_4 plants. Cole and Monger (1994(a)(b)) have proposed that a post-glacial increase in atmospheric CO_2, from about 180 to 270 p.p.m.v., directly contributed to the shift from C_4 to C_3 vegetation in the Chihuahuan desert, New Mexico, USA.

Although C_3 and C_4 plants require different concentrations of atmospheric CO_2 for photosynthesis, it is debatable as to whether a shift of this nature would cause a change in the isotopic composition of the plant community (Boutton *et al.* 1994). It is more likely that other environmental factors such as those discussed above would be more important in determining changes in the distribution of plants.

Transferral Of $\delta^{13}C$ Values To Terrestrial Fauna

Vegetation is seldom directly preserved in deposits, and therefore, must be indirectly identified through other means. Experiments in laboratory and field situations have shown that the $\delta^{13}C$ values of skeletal tissues of animals reflect the isotopic composition of food consumed (DeNiro and Epstein 1978; Vogel 1978; van der Merwe 1982; DeNiro and Schoeninger 1983; Tieszen *et al.* 1983; Hobson and Schwarcz 1986; Ambrose and Norr 1993). A regional comparison of $\delta^{13}C$ values of modern and archaeologically recovered ungulate grazers provides an opportunity to document shifts in C_3 and C_4 *grasses* over time, whereas browsers will reflect changes in forest canopy and provide a $\delta^{13}C$ baseline for a C_3 environment. This in turn allows for inferences to be made about palaeoenvironmental patterns that influence plant type distribution and animal habitat utilization.

Experiments have shown that in relation to the source vegetation the whole animal is enriched by 1‰, and that individual tissues are predictably patterned with regards to the source (DeNiro and Epstein 1978; Vogel 1978; Tieszen 1991). There is a consistently significant difference between the $\delta^{13}C$ values of ungulate herbivores occupying different vegetational habitats, as well as between various tissues (collagen, skin, muscle, hair, fat, and biological apatite) from individual ungulates (DeNiro and Epstein 1978; Vogel 1978; Tieszen 1991; Lee-Thorp 1989; Lee-Thorp *et al.* 1989). The latter internal fractionation may be the result of isotope effects inherent in the conversion of dietary carbon to tissue carbon, and the partitioning of dietary carbon for synthesis of different tissues (Krueger and Sullivan 1984; Lee-Thorp *et al.* 1989; Tieszen 1991; Ambrose and Norr 1993). The long term preservation of carbon in collagen (several thousands of years) and biological apatite (several millions of years) makes them suitable sources for tracing the dietary patterns and environments of herbivores through time.

Stable Carbon Isotopes In Calcified Tissues Of Terrestrial Fauna

Collagen

Collagen is a structural protein formed in bone, tooth

dentine, skin, and muscle tissues, and constitutes 85 to 90% of the organic matrix of calcified tissues. In relation to whole bone, the organic matrix contributes 30% of the weight; the remaining 70% being made up by the inorganic phases (Hare 1980; Price 1989). Collagen protein is distinguished from other endogenous and exogenous proteins by its triple helix shape and unique amino acid composition characterized by the presence of glycine, proline, hydroxyproline, and hydroxylysine. The term "collagen" as used here, and by most other researchers in this field, refers to acid-insoluble protein residue. It includes small amounts of non-collagenous proteins and is thus not entirely equivalent to collagen as understood by biochemists.

Controlled eating experiments conducted on small laboratory animals showed that there is an enrichment in ^{13}C in bone collagen by 3 to 4‰ compared with food eaten (DeNiro and Epstein 1978; Tieszen *et al.* 1983). In contrast, large free-ranging ungulate herbivores sampled in the field had a diet to collagen difference of 5‰. This discrepancy may be related to: 1) differences in metabolic and assimilation rates between smaller short lived laboratory animals and those of large ungulates with longer lifespans, and 2) differences in quality of diet, most importantly the amount and source of protein (Tieszen 1991:240; Ambrose and Norr 1993:7). With regards to the latter point, collagen is formed

primarily from dietary protein, but when protein is insufficient, carbon will be synthesized from carbohydrates in the diet. As proteins are depleted in ^{13}C in relation to carbohydrates, the constantly higher protein diet of the laboratory animals, versus the low quality grasses most often processed by herbivores in the wild, may account for the discrepancy between the two experimental groups (Tieszen 1991:24). A point for further consideration is that the protein portion of the laboratory diets often have different $\delta^{13}C$ values from those of the free-range diets (Ambrose and Norr 1993) .

Collagen in bone is continually formed and reworked over the life span of an individual. The time that it takes for bone collagen to replace itself is not clearly understood, as it depends on several variables including health, age, and nutritional status of an individual, and the skeletal element analysed. Reports frequently cite turnover rates to be anywhere between 10 years (Libby *et al.* 1964) to 30 years (Stenhouse and Baxter 1976, 1979), with adults and cortical bone having slower rates than juveniles and cancellous/ trabecular bone (Libby *et al.* 1964; Klepinger 1984). Thus, bone collagen will represent a medium to long-term dietary average and obscure short-term trends (Chisholm 1989:21).

In contrast, collagen in tooth dentine is not extensively reworked after tooth formation. There is some addition of secondary and possibly tertiary dentine in later life, but overall, $\delta^{13}C$ values primarily reflect diet during tooth formation (Sealy *et al.* 1995)

Biological Apatite

The inorganic (mineral) phase of calcified tissues consists primarily of calcium phosphate crystallized into a structure analogous to hydroxyapatite $\{Ca_{10}(PO_4)_6(OH)_2\}$. As the structure is characterized by a variety of ion substitutions, it is more appropriately referred to as biological apatite (or apatite). The crystal structure is hexagonal in shape and small in size (Jackson *et al.* 1978; LeGeros 1981). Carbon is deposited in the biological apatite structure of bone and teeth as carbonate ions (CO_3^{2-}) substituting for phosphate ions (PO_4^{3-}), or by adsorption of CO_3^{2-} and possibly bicarbonate (HCO_3^-) (Betts *et al.* 1981; LeGeros 1981). Dietary carbon is represented by carbonates derived from blood bicarbonates.

Herbivores in laboratory feeding studies and field observations demonstrated enrichment in apatite $\delta^{13}C$ values compared to the dietary source of 9.5 to 10‰, and 12‰ respectively (DeNiro and Epstein 1978; Lee-Thorp and van der Merwe 1987; Lee-Thorp 1989; Ambrose and Norr

1993). Enrichment has been attributed to fractionation between CO_2 and the formation of HCO_3^- and CO_3^{2-} (Lee-Thorp 1989:48).

A comparison of $\delta^{13}C$ values of various tissuses from individual herbivores established that ^{13}C in apatite is enriched approximately 7 to 8‰ compared with that of collagen (Sullivan and Krueger 1981; Lee-Thorp 1989; Ambrose and Norr 1993). This enrichment may be due to the more uniform incorporation of all dietary sources into apatite, whereas collagen is formed mostly from dietary protein (Ambrose and Norr 1993:29).

As with collagen, apatite is deposited and remodelled in bone throughout the life of the individual, but it is unclear whether the turnover rate is similar to that of collagen. In teeth, apatite is found mainly in the enamel component, and has $\delta^{13}C$ values that reflect the diet during enamel formation. It is assumed that diet and habitat of the herbivores analysed in this study have not changed significantly over their lifetime; thus, bone and teeth will reflect a similar diet.

Diagenesis

Diagenesis refers to the chemical and/or physical alteration in the original composition of a material during and after deposition. It is important to be able

to identify any loss, addition, or exchange of material that could alter the original dietary isotopic signature. Diagenetic processes responsible for the modification of collagen and biological apatite are highly variable from one environment to another. They depend on a range of biological, chemical, and physical factors which include: time and temperature (Hare 1980; Nelson *et al*. 1986; Child 1995; Hedges *et al*. 1995); microbial activities (Grupe *et al*. 1993; Child 1995); groundwater erosion (Krueger 1991; Hedges and Millard 1995); depositional matrix, and humic uptake (Grupe 1995; van Klinken and Hedges 1995). Optimal conditions for preservation of calcified tissues tend to be rapid burial in slightly alkaline deposits and/or cool dry anoxic environments, which have limited amounts of microbial and groundwater activities, and humic formation (Child 1995; Grupe 1995; Hedges and Millard 1995; van Klinken and Hedges 1995).

Collagen isotopic ratios are fairly robust, but if the collagen structure uncouples there may be preferential loss of particular amino acids. This need not pose a problem, for as long as the appropriate amino acids are present the original $^{13}C/^{12}C$ ratio should be intact (Hare 1980; Masters 1987; Grupe 1995). Likewise, exogenous carbon, such as humates or non-collagenous proteins, can be removed by proper chemical pretreatment (Sealy 1986; Chisholm *et al*. 1983). Collagen protected in dense

cortical bone and teeth is preferred for analysis, as it is less open to contamination and deteriorates more slowly than collagen found in porous trabecular/ cancellous bone.

Various methods have been proposed to determine whether the appropriate collagen proteins have been suitably preserved. The presence of certain amino acids, in particular hydroxyproline, has been cited as a reliable indicator of collagen preservation (Chisholm 1989; Hare *et al.* 1991). Grupe *et al.* (1993) have cautioned that hydroxyproline may not be a suitable marker. They recommend monitoring the presence of glycine, which is more abundant in collagen, but smaller in size and, therefore, less susceptible to microbes. Due to expense, amino acid testing is not always feasible. More economical means are to observe that structural integrity of the sample has been maintained (Schoeninger *et al.* 1989), and determination of an acceptable carbon to nitrogen (C/N) ratio for collagen (DeNiro 1985). Archaeological collagen with a C/N ratio within the range of modern collagen, 2.9 to 3.6, is likely to yield reliable isotope ratios (DeNiro 1985).

Unlike collagen, where the isotopic composition remains fairly intact as long as enough collagen remains, the original isotopic signature of apatite is more prone to alterations from the addition of exogenous carbonate,

ionic and isotopic exchange, and recrystallization. To limit the potential for diagenetic influences, analysis of enamel apatite is preferred over bone apatite. In enamel, apatite is more crystalline and stable, and denser and less porous than bone, thereby, minimizing decomposition, crystal growth, and exogenous contaminates.

Where exogenous carbonates are precipitated into the apatite matrix from carbonates in groundwater, these can be easily removed by appropriate acidic pretreatment (Sullivan and Krueger 1981; Lee-Thorp 1989; Lee-Thorp *et al*. 1989; Krueger 1991). However, in some instances, the original isotopic ratio may be erased as a result of ionic and isotopic exchange.

Recrystallization is a process whereby apatite crystals are dissolved and reformed, usually into larger and more stable configurations. During this process exogenous carbonates may be incorporated. As fossilization progresses apatite becomes more crystalline, but this process, and possible alteration of the isotopic signature, may take several thousand years (Lee-Thorp 1989; Sillen 1989).

Although recrystallization usually occurs to some extent, Lee-Thorp (1989:32) suggests that the effect on the original isotopic signature "will be minimal if

recrystallisation merely involves a rearrangement of the biogenic constituents into larger crystals". If minimal, biogenic apatite may be retrieved through a series of dilute acetic acid washes, as it is more soluble than recrystallized apatite, but less soluble than diagenetic carbonate (Sillen 1989). The extent of recrystallization and contamination can be assessed through the use of x-ray diffraction, infrared spectroscopy, nuclear magnetic, and electron spin resonance spectroscopy (Lee-Thorp 1989; Sillen 1989; Lee-Thorp and van der Merwe 1991; Lee *et al*. 1995; Rink and Schwarcz 1995).

Stable Carbon Isotopes In Soil Sediments

Soils form from the *in situ* chemical and physical weathering of a parent rock material to produce sediments that can support plant growth and contain the remains of organic materials. Sediments that have been transported physically or chemically from one location to another may also become associated with a soil profile. In soil, sediments are divided vertically into zones of variable thickness comprising mineral and/or organic compounds, which may differ from the parent rock in morphological, physical, chemical, mineralogical, and biological characteristics (Birkeland 1984:3). Horizons are formed by the vertical movement and accumulation of materials, principally minerals and organic matter,

within the profile. From the surface downward, horizons are characterized by Birkeland (1984:6) as:

O Horizon - surface accumulation of fresh and/or partly decomposed organic matter; may or may not occur in waterlogged conditions.

A Horizon - high accumulation of organic matter mixed with minerals; zone of eluviation.

E Horizon - (not always present) - underlies O or A horizon and contains less of one or all of the following than the underlying horizon: organic matter,clay, and sequoixides (iron, aluminum, and oxides); accumulation of quartz and/or sands, or silts.

B Horizon - a zone of illuviation, accumulation, and/or alteration of one or all of the following: silicate clays, sequoixides, and organic matter.

C Horizon - weathered but intact bedrock or parent rock material, which may or may not be similar in characteristic to the soil sediments.

More than one soil forming event may have taken place on a landscape. A palaeosol, or an older soil profile, may remain at the surface following the soil formation process, or be buried under more recent sediment or soil deposits. After burial, a palaeosol may once again be exposed due to erosion of overlaying material.

Soil CO_2 is derived primarily from the decomposition of soil organic matter (SOM) and plant respiration. Decomposed SOM is preserved in microbial metabolites and cell walls, and in organo-mineral complexes associated with soil sediments (Bruckert 1982). The relative proportion of C_3/C_4 plant growth on a soil surface should

be reflected in the $\delta^{13}C$ values of the SOM found in these components (O'Brien and Stout 1978; Schwartz *et al.* 1986; Guillet *et al.* 1988; Martin *et al.* 1990; Bond *et al.* 1994). However, for this to take place certain conditions need to have been operant (Dzurec *et al.* 1985:17; Tieszen and Boutton 1989:188). First, there must have been no significant or inconsistent fractionation of soil plant carbon as a result of either preservation or diagenetic processes. Secondly, the relative proportions of C_3/C_4 vegetation must have remained stable for a period equal to that of the oldest SOM in the profile. If the second premise does not hold, then there should be a difference in the $\delta^{13}C$ values between SOM and the existing vegetation, suggesting a change in the abundance of C_3/C_4 vegetation over time.

Organic matter may also enter a deposit by being transported to a location, and may therefore, not represent *in situ* developments. This is an important consideration when dealing with archaeological deposits where plants and grasses have been brought into sites by people, and external sediments have accumulated through various processes (Stein 1992:192-196).

Isotopic Fractionation In Soil Sediments

The principles of plant decomposition and the

incorporation of $^{13}C/^{12}C$ into soil sediments have been outlined in various texts (see Deines 1980). When a plant dies, its organic compounds become unstable and begin to decompose, at which point micro-organisms attack and break down biopolymers, proteins, and nucleic acids to soluble compounds (Fig. 2.3). From this, humus, the completely decayed residues and cellular fractions of SOM, is formed. Humus makes up the bulk of the SOM and is defined as,

> a complex and rather resistant mixture of brown and dark brown amorphous and colloidal substances modified from the original tissues or synthesized by the various soil organisms (Buckman and Brady 1969:653).

Humus is subdivided into three fractions based on solubility: 1) humins - insoluble at basic pH; 2) humic acids - dark in colour and soluble at basic pH, but precipitating at pH 1, and 3) fulvic acids - yellow to red in colour and soluble in both acidic and basic solutions. Fulvic acids are more enriched in ^{13}C, humins intermediate, and humic acids slightly depleted. The isotopic depletion of humic acids compared to fulvic acids is attributed to the loss of a ^{13}C-rich carboxyl group during the humification process (Deines 1980:346).

Differential decomposition of plant biochemical fractions in the SOM shows slight variation in relation to the carbon isotopic composition of the source

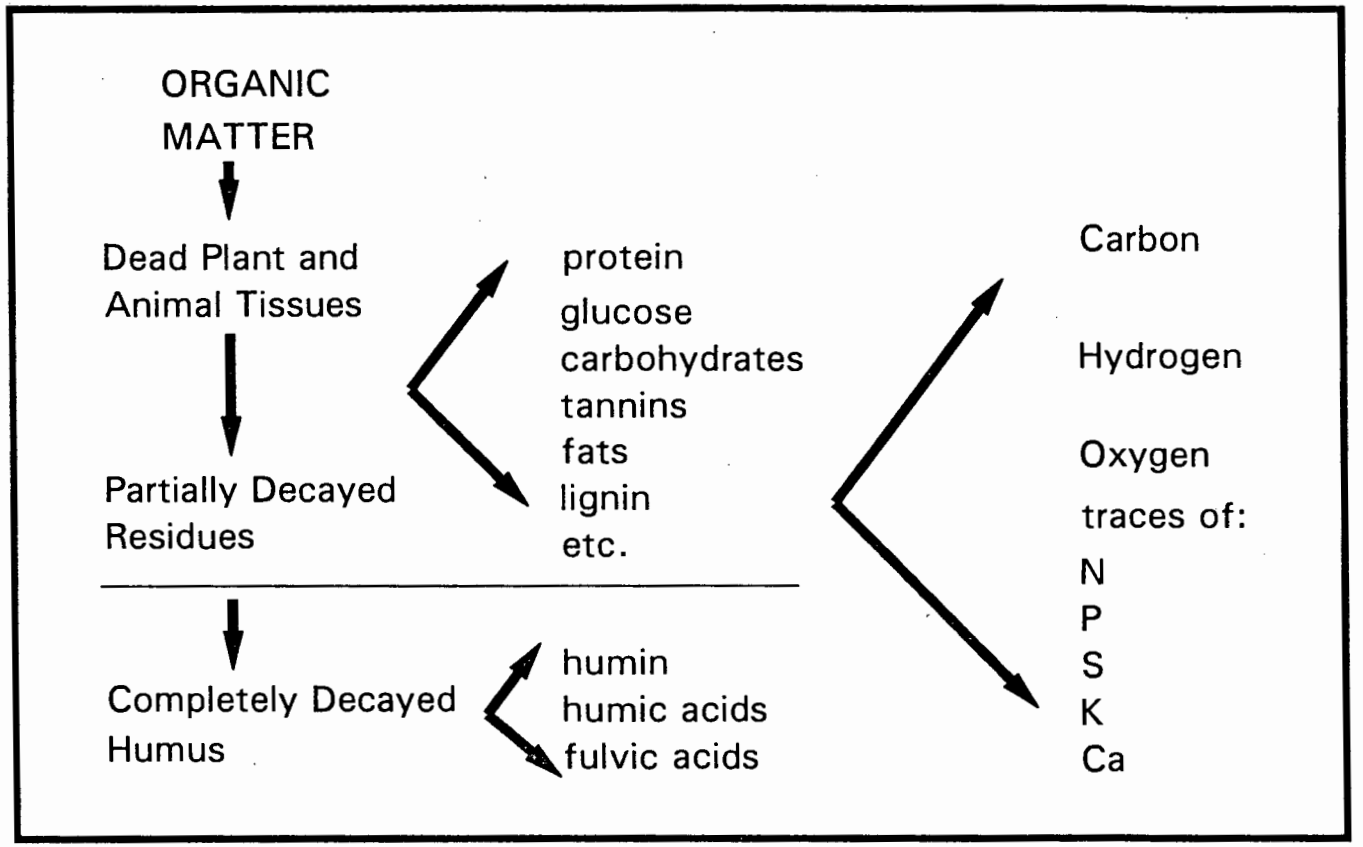

Figure 2.3 Decomposition of organic matter and resulting products (redrawn from Stein 1992)

vegetation. Biochemical fractions such as starch and cellulose are enriched in ^{13}C by 1 to 3‰ compared with whole plant tissue (Smith and Epstein 1971). This fractionation is negligible compared to the 14‰ difference between C_3 and C_4 vegetation types. Exceptions to this are plant lipids, which are consistently depleted in ^{13}C by 5 to 10‰ compared with the whole plant (Smith and Epstein 1971). However, they are usually removed from sediments within one year after deposition (Dzurec *et al.* 1985:18).

Further studies have demonstrated that the $\delta^{13}C$ values of SOM are enriched by 1 to 3‰ with increased depth of the soil profile, suggesting a certain amount of fractionation during decomposition (Rosenfeld and Silverman 1959; Stout *et al.* 1975; O'Brien and Stout 1978; Dzurec *et al.* 1985; Natelhoffer and Fry 1988). This fractionation has been attributed to microbotic activities. Micro-organisms utilize $^{12}CO_2$ in preference to $^{13}CO_2$, leading to slight ^{13}C enrichment of the residual organic substrate (Rosenfeld and Silverman 1959). In contrast, there appears to be little or no carbon isotopic fractionation in soils where plant decomposition is restricted and undecomposed plant material accumulates (Dzurec 1985; Tieszen 1991).

In arid, semiarid, or alkaline environments soil carbonate nodules may form from soil CO_2 sources (Cerling

1984). Fractionation during nodule formation leads to a 14 to 17‰ enrichment in ^{13}C compared to contributing vegetation and SOM. Any change in this net difference is attributed to diagenetic alteration of the carbonate nodule (Cerling *et al.* 1989).

Turnover Rates Of Soil Organic Matter

During the development of a soil horizon, organic matter previously deposited is added to by more recently decayed organics. In order for stable carbon isotopic studies to reflect past vegetation composition it is necessary that previous signatures are not masked by subsequent changes in the vegetational community, thereby creating a homogeneous profile. Studies indicate that rates of change and preservation of soil ^{13}C properties can be obtained by measuring the isotopic composition of SOM in various soil particle size fractions (Anderson *et al.* 1981; Dzurec *et al.* 1985; Balesdent *et al.* 1988). Once identified, points of turnover in the substrate can be dated directly through radiocarbon dating or indirectly from particle size fractions and depth correlations in the soil profile.

Controlled studies have been carried out on the turnover rate of natural C_3/C_4 vegetation in soil. These studies entailed replacing an established vegetation type with another and measuring the turnover rate (Balesdent

et al. 1988; Vitorello *et al.* 1989; Martin *et al.* 1990; Skjemstad *et al.* 1990). The data showed that clay size fractions retain the original SOM $\delta^{13}C$ signature for a longer period of time than larger particles, and that decomposition of organic material does not proceed at a constant rate. There is rapid replacement by the vegetation in the first 20 years. After this point, the rate decreases as all organic matter associated with the larger sediment particles has been replaced by the new vegetation, leaving only the more stable SOM in the clay fractions (Skjemstad *et al.* 1990:272). Stability of this nature has been demonstrated by Balesdent *et al.* (1988) on agricultural systems in which, after one hundred years of C_3 cultivation, $\delta^{13}C$ values representing the previous C_4 prairie grassland still accounted for more than fifty percent of the total soil organic content. In shallow soil profiles, analysis of different size particles can aid in identifying the previous vegetational composition. In South Africa, Stock *et al.* (1993) used this approach to determine the isotopic signature of vegetation in the Western Cape prior to European settlement.

Radiocarbon dating used to measure organic turnover rates indicates that SOM can have a mean residence time ranging from several hundred to several thousand years (O'Brien and Stout 1978; Trumbore *et al.* 1989). However, dating of whole organics gives a weighted mean

for the periods in which the older fraction decayed and younger organic matter accumulated in the profile (Botha *et al*. 1992:509; Stein 1992:202,203). Separation of humus into fulvic acids and humic acids offers a potential means of isolating SOM of varying ages (Alessio *et al*. 1989; Trumbore *et al*. 1989; Matthews 1993). Fulvic acids are highly mobile fractions containing the youngest soil organics and tend to accumulate migratory contaminants causing unreliable results. Humic acids are less mobile, contain fewer contaminants, and retain relatively older soil organics. Radiocarbon dates on biochemical fractions from buried soil profiles show that fulvic acids tend to be significantly younger than humic acids from the same sample (Matthews 1993). Fulvic acids reflect the last vegetation to decay prior to burial of the soil profile; offering an estimated maximum age since the burial of the soil horizon. Humic acids preserve old carbon within the buried soil; providing the estimated minimal length of time since the onset of soil formation.

Conclusion

The distinct isotopic composition and physical requirements of C_3 and C_4 vegetation provide the basis for studying past environmental and climatic conditions, as reconstructed from the calcified tissues of ungulate herbivores and soil organic matter. Studying modern

analogues provides an understanding of the environmental and metabolic factors that contribute to the $\delta^{13}C$ values of plants and the transferral of that signature through the trophic levels. In order to have a baseline from which to measure past changes, it must be assumed that these factors have remained relatively constant through time. Nevertheless, it is important to keep in mind that ecosystems are non-static. All possible influences, natural and anthropogenic, need to be considered before interpretations can be made; particularly when drawing comparisons from previous research.

CHAPTER 3

LABORATORY PROCEDURES

The methods used to covert a sample to CO_2 in order to determine stable carbon isotopic ratios by mass spectrometry, are discussed in this chapter. **Inorganic carbon** - carbonate in enamel apatite - and **organic carbon** - bone and dentin collagen, soil organic matter, and plant material - have been used as sample materials for the purpose of environmental reconstruction. The different methods of preparation and analytical techniques required for these materials are examined in order to evaluate the advantages and limitations of each and the reliability of the $\delta^{13}C$ values produced.

Preparation of Samples

Techniques used to isolate the carbon component of a sample for isotopic analysis and convert it to CO_2 will vary depending on the matrix in which it is contained and the type of mass spectrometer used. It is important that these techniques do not cause isotopic fractionation.

Plants

Of the materials analysed, plants require the simplest form of preparation. Surface contaminants were cleaned

from the dry plant, and using tweezers and scalpel approximately 0.050mg of plant material was removed and placed into a foil cup. Small scale measurements were made possible through the use of a electronic microbalance. The foil cup was folded closed to secure the sample and prevent contamination from particles that could affect the isotopic reading.

Collagen

Collagen has commonly been extracted using one of two methods. In both cases acid insoluble collagen protein is isolated by removal of minerals and exogenous organic materials from the bone and dentine matrix. In the gelatin method (Longin 1971; Chisholm *et al.* 1983; Chisholm 1989), bone or dentine powder is demineralized in an acid solution, followed by treatment with a sodium hydroxide (NaOH) solution to remove humics. Collagen is solubilized through slow hydrolysis in a hot dilute acidic solution and filtered to remove remaining impurities. The filtrate is then evaporated to recover the dissolved "gelatin".

The second technique of collagen extraction was adapted from methods originally developed by radiocarbon chemists, and is most commonly used in the University of Cape Town's (UCT) Archaeometry Laboratory (Sealy 1986; Lee-Thorp 1989). Bone and dentine samples in this study

were prepared according to this method.

Dentine was separated from tooth enamel by using hand held pliers and rotary blade drill. The surfaces of bone and dentine fragments were cleaned manually with brush and distilled water, and then sonicated in distilled water to remove subsurface dirt. When using bone or dentine chunks there is potential for rootlets to be trapped in the subsurface structure of the sample. However, rootlets not removed during cleaning or chemical treatment are detectable when the sample is sectioned for analysis. Between 0.20 and 0.40g of clean dry sample was weighed (using an electronic balance) and demineralized in a beaker containing 1% hydrochloric acid (HCl). Depending on the size and density of the sample, this process may take from a few days to about 2 weeks to complete. The 1% HCl solution was replaced every second day until gases stopped evolving and the sample was translucent. At this point the sample was a flexible isomorph replicate of the original bone or dentine structure. After rinsing to neutrality (pH 6) with distilled water, humates were removed by immersion in a 0.125M NaOH solution for 24 hours. Samples were again rinsed in distilled water until all traces of NaOH were removed. For small samples, NaOH treatment lasts for only 1 hour. Humates were precipitated from the NaOH supernatant with 1 to 2ml of concentrated HCl. Finally, both collagen and humates were freeze-dried.

Schoeninger *et al.* (1989) found that the simplicity of the isomorph method does not undermine the carbon composition of collagen, as amino acid profiles produced are consistent with unaltered collagen. Any separation of peptides and amino acids from collagen protein is usually apparent in the visible deterioration of the morphological integrity of the isomorph. This separation is not visually recognizable in gelatin. Such alterations in collagen protein are important as they may result in a biased $\delta^{13}C$ value (Hare *et al.* 1991). One problem facing both the isomorph and gelatin method is that HCl may result in loss of organic material and NaOH may affect isotopic character and diminish collagen yield (Chisholm *et al.* 1983; Chisholm 1989; van Klinken and Hedges 1995). These changes are not considered significant enough to warrant the disuse of either chemical pretreatment, as it is more important to eliminate minerals and humates.

Lipids in modern and fatty samples need to be removed, as they are more depleted in ^{13}C than protein and would bias results (Chisholm *et al.* 1983). In this study it was felt that lipid removal was not required as lipids are rarely preserved in archaeological samples.

For this study two criteria were used to determine collagen purity. One was the presence of an isomorph that is pale yellow to white in colour. A brownish

colour isomorph indicates possible contamination by humates. In a C_3 environment or a pure C_4 grassland, uncontaminated collagen is more enriched in ^{13}C than humates by approximately 5‰, since humates should reflect the vegetational source. The second criteria for collagen purity used was a C/N ratio between 2.9 and 3.6 (DeNiro 1985). Although these criteria are, by biochemical standards, fairly crude, extensive testing in the UCT Archaeometry Laboratory has demonstrated that samples that satisfy them generally yield reliable isotopic values.

Apatite

Isotopic analysis of apatite requires that the biogenic calcium phosphate structure be separated from the organic phase and contaminants. Inorganic contaminants of relevance consist mainly of diagenetic carbonates (Lee-Thorp 1989; Sillen 1989; Lee-Thorp and van der Merwe 1991). Until relatively recently debate existed about ways of pretreating the mineral phase and validity of these methods to remove contaminates (Land *et al.* 1980; Sullivan and Krueger 1981; Schoeninger and DeNiro 1982; Sullivan and Krueger 1983). Utilizing techniques employed by geochemists for carbonate extraction, studies undertaken by Sullivan and Krueger (1981) and Lee-Thorp (1989) have shown that with correct pretreatment reliable isotopic values can be obtained.

Samples were recorded, cleaned, and enamel separated from dentine as outlined for collagen preparation. Clean enamel is broken into small pieces, and ground to a powder in a liquid nitrogen cooled Spex mill. To remove organic matter, enamel powder was weighed into a 50ml centrifuge tube and treated with 40ml of approximately 2% sodium hypochlorite solution (NaOCl). Initially between 1.25 to 1.50g of the enamel powder was used for apatite extraction, but later experiments determined that approximately 0.50g of enamel produced sufficient apatite for CO_2 production. This latter amount was then subsequently used. Samples were left in NaOCl solution for 1 to 2 days and stirred frequently. Once the reaction ceased, the powder was alternately washed with distilled water and centrifuged until neutral. 40ml of 0.1M acetic acid (CH_3COOH) was added to the anorganic powder and stirred frequently in order to remove diagenetic carbonates. The reaction took up to 3 days to reach completion, when visible reaction ceased. In previous accounts (Lee-Thorp 1989; Lee-Thorp *et al.* 1989) 1M acetic acid was used, but the tooth enamel processed in this project had minimal carbonate contamination and a less rigorous acid treatment was preferred. The acid treated apatite was washed and centrifuged until neutral, and freeze-dried.

Unlike collagen, there is no visual means to assess whether contaminants have been fully removed from the

apatite sample. For this study, when material was available, a collagen-apatite difference of approximately 8‰, and a comparison of $\delta^{13}C$ values between herbivores with isotopically distinct diets, in this instance grazers and browsers from the same stratigraphic level, provided means of evaluating the validity of the isotopic values for apatite.

Soil Organic Matter

For soil organic matter (SOM) two methods of preparation have been used in this study. Each method produces both contaminant-free whole soil particles and comparable isotopic results. The two methods differ in scale with regards to sample size, chemical treatment, and preparation time. The first method is that used in the UCT Botany Department (Bond *et al.* 1994; Stock pers. comm.). Sediments were air-dried in the laboratory. Large aggregates were broken up and sediments were sieved through <200μm mesh to remove stones and large organic fragments. 200g of sieved sediments was added to 1 litre of distilled water and stirred. All organic matter floating to the top was removed, and a portion sampled for analysis.

Sediments containing micro-organic fractions were oven-dried at 100°C, after which all aggregates were crushed into uniform consistency. From this a subsample

of 100mg of sediment was placed into a small Vycor quartz tube (subsequently referred to as a quartz boat). The remaining sediments were set aside for future analysis. When weighing out the subsample any visible organic matter was discarded. To dissolve soil carbonates 0.2μl of concentrated HCl was added to the 100mg subsample. The quartz boat was placed inside a larger Vycor quartz tube and oven-dried at 120°C for 2 to 3 days until HCl had evaporated. The oven was checked periodically to ensure that the temperature was not too high, and the HCl solution had not boiled over.

The second method for preparing soil organics from this study follows that outlined by Cerling *et al.* (1991), with further notes from Ambrose (pers. comm.). This method produced a larger purified sample, and was less time consuming and labour intensive. Samples processed using both methods produced comparable results. All aggregates of sediments were broken up and macro-organic matter removed. Sediments were sieved through <200μm mesh. The macro-organic matter was treated in a manner similar to the previous method. A 10g subsample of sieved sediments containing micro-organics was placed in a 50ml centrifuge tube and carbonates dissolved with 40ml of 1M HCl at a room temperature of 25°C. To ensure that the carbonates were completely dissolved, the samples were frequently stirred and the 1M HCl solution regularly changed until no further reaction was

observed. The sediments were alternately rinsed with distilled water, centrifuged until pH 3 was reached, and freeze-dried.

CO_2 Production

After isolating organic (collagen and soil organics) and inorganic (apatite) carbon from the sample matrix, it must next be converted to CO_2 gas, which can be analysed by mass spectrometry. Different methods of CO_2 production were employed for organic and inorganic materials, and plants.

Organic CO_2

The following were the approximate sample sizes used for the purpose of CO_2 production:

Collagen - 10mg
Sediments - 100mg
Ground Macro-Organic Matter - 5mg.

Conversion to CO_2 took place through off-line combustion, as described by Sofer (1980) and Sealy (1986). Vycor quartz combustion tubes were prepared and baked at 450°C for 2 hours to remove organic contaminants, for example machine oils. Samples were weighed and loaded directly into the cleaned tubes. Sediments were loaded first

into quartz boats to prevent any small particles adhering to the side of the tubes and/or remaining alkali substances from reacting with the quartz combustion tubes and causing them to crack. Sediments processed according to the first method were already in the appropriate combustion tubes. Approximately 1.0g of wire-form cupric oxide (CuO), 1.0g of copper metal (Cu) and a small piece of clean silver (Ag) foil were added to the tubes, which were evacuated on a vacuum line to $<10^{-2}$ Torr and sealed. Combustion of the sealed samples took place in a furnace heated to 800°C for 6 hours, then allowed to slowly cool for about 24 hours. The end result of combustion was that CuO was reduced, oxygen combined with organic carbon to form CO_2, while cooling Cu reduced NO_x to N_2. Any halogens and sulphur were removed through reaction with Ag. The resulting CO_2, N_2, and water vapour (H_2O) were separated cryogenically and purified soon after combustion to avoid the gases reacting with any metallic copper present, thereby necessitating re-combustion (Stuiver *et al.* 1984).

Inorganic CO_2

To convert CO_3^{2-} (apatite carbonate) to CO_2 the method originally outlined by McCrea (1950), as adapted by Lee-Thorp (1989) was followed. Between 125 and 150mg of apatite was placed into the main body of an inverted y-shaped reaction vessel. Approximately 3ml of 100%

phosphoric acid (H_3PO_4), kept dry under vacuum, was introduced into the arm of the vessel. An adaptor was fitted onto the vessel, which was then evacuated on a vacuum line to $<10^{-2}$ Torr. Once the acid had been fully evacuated and degassed (no bubbles forming in the acid), the adaptor was closed, the vessel was placed into a constant temperature waterbath of 25°C and allowed to equilibrate. After 30 minutes the acid was tipped onto the apatite. Although most of the reaction takes place fairly rapidly, approximately 3 to 4 days was usually needed to reach completion. During hydrolysis CO_2 and H_2O were formed.

CO_2 Purification

Collagen, Apatite, And Soil Sediments

Cryogenic separation and purification of gases took place on a vacuum line evacuated to 10^{-4} Torr. For organic samples the combustion tube was scored and placed into a flexible metal "cracker" attached to the vacuum line. The gases were released by flexing the cracker and breaking the tube. The reaction vessel used for inorganic samples was inserted into an O-ring coupling and gases released into the evacuated line by opening the adaptor. Gases were separated (purified) by cryogenic distillation using first liquid nitrogen to freeze out CO_2, followed by ethanol-dry-ice to freeze out

H_2O vapour. Purified CO_2 gas was cryogenically transferred and collected in a Pyrex glass tube.

Plants

Plant samples were combusted in an on-line NA 1500 Carla-Erba C/N elemental analyzer, where combustion and purification are linked to the mass spectrometer. The foil cup containing plant material was placed into a carousel that introduces it into the gas purification system. Inside the system the sample was flash-combusted at a temperature of between 1600 and 1800°C in an oxygen stream. The resulting gases were carried in a helium stream through a series of temperature-controlled cryogenic traps where CO_2, N_2, and H_2O were collected. Once separated, CO_2 was admitted into a dual-inlet isotope ratio Finnigan MAT 252 mass spectrometer for analysis. This procedure, though automated, is similar to the conventional off-line preparation and manual introduction of sample to the dual-inlet mass spectrometer, which was used for all other samples in this study.

Mass Spectrometry

Most measurements reported in this thesis were carried out on a VG Micromass 602E dual-inlet isotope ratio mass spectrometer, designed to measure isotope ratios in

light elements (mass numbers ca. <64). A sample gas was introduced via a cracker system connected to the mass spectrometer, and broken under a vacuum of 10^{-3} Torr. Small CO_2 samples, such as those often obtained from soils or sediments, were first frozen into a cold finger to concentrate sample into a smaller volume, and then introduced into the mass spectrometer. The dual-inlet design allows the sample gas and the reference gas, with a known isotopic ratio, to be admitted to the instrument alternately. Gases were ionized by a hot filament and accelerated, under a vacuum of $\pm 10^{-8}$ Torr, through a magnetic field that dispersed and measured the ions according to light and heavy isotopes (Fig. 3.1). Repeated comparisons of the ratio $^{13}C/^{12}C$ in the sample gas with that in the reference gas allowed detection of very small variations in the relative isotope abundance between the two.

The laboratory CO_2 reference gas was calibrated against NBS standards 16, 17, 19, and 20. These standards have isotope ratios that were well characterized in relation to the internationally accept Chicago *Peedee belemnite* (PDB) marine limestone standard, which has a $\delta^{13}C$ value of 0‰.

The resulting isotope abundances are expressed as $\delta^{13}C$ values. Based on repeated measurements of the same sample gas, a mass spectrometer precision of better than

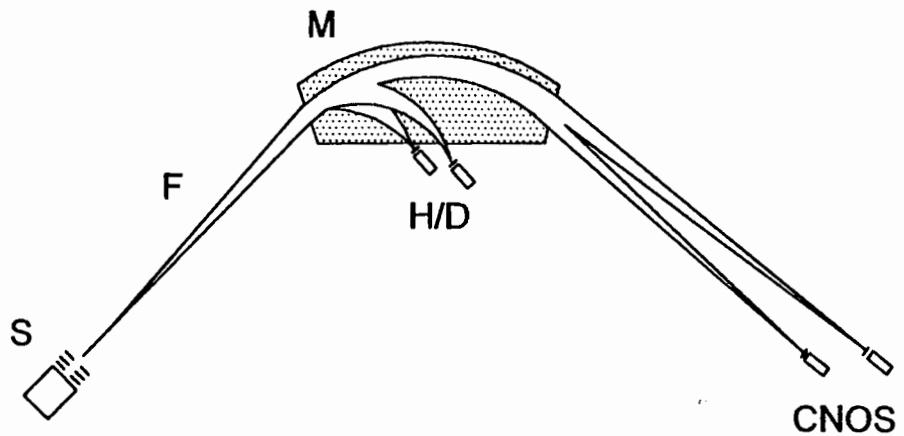

Figure 3.1 Components of an isotope ratio mass spectrometer: ionizing source (S), flight tube (F), magnet (M), Faraday cups for detecting hydrogen isotopes (H/D), and Faraday cups for detecting carbon, nitrogen, and sulphur (S) (redrawn from Ehleringer and Osmond 1991)

0.1‰ is expected for stable carbon isotopic values obtained in this study.

Conclusion

An outline of laboratory procedures used in this study has been provided. The advantages and limitations of these methods have been reviewed in relation to the detection and removal of contaminants, production of CO_2, and mass spectrometry. Overall, these methods have proven to be successful in yielding reliable isotopic results.

CHAPTER 4

THE MODERN GRASSLAND BIOME

There are many ecosystems that work together to shape an environment. These ecosystems are made up of interlinking biotic and abiotic factors and cannot be properly understood through a single component alone. It has been argued that the underlying cause of change in an ecosystem is climate and consequently changes in the composition of the soils, plants and animals in a particular community will reflect climatic change (Ellery *et al.* 1991; Rutherford and Westfall 1994). And yet, the effect of humans as a part of the environment, especially with the arrival of pastoralists, agriculturalists, and Europeans over the last 2000 years in southern Africa cannot be ignored. Environmental modification, which may be misconstrued as climatic in origins, has occurred as result of human exploitation and management of natural resources, and the introduction of exotic plants and animals.

The modern environment of the study area, the Caledon River Valley, is examined to provide a starting point from which to identify underlying causes of change. It should be noted, however, as Hill (1994:323) emphasises,

> that fossil assemblages are quite different from modern living communities...we need to examine the modern world from the point of view of the fossil record, rather than vice versa.

The Study Area

The Caledon River is situated along the international border between the eastern Free State and western Lesotho, with the river valley forming a 50km wide belt from the town of Clarens in the northeastern Free State to the Orange (Senqu) River in the southwest. Within this belt the sites studied are situated in the local areas of: Ladybrand and Marquard to the west of the river, and the Roma Valley and the Phuthiatsana-ea-Thaba Bosiu (**PTB**) River Basin (Fig. 4.1). In general, the geology, geomorphology, climate, soils, vegetation, and fauna of these four localities fall within the sub-humid, climatic climax grasslands of the Grassland Biome of southern Africa (Mentis and Huntley 1982; Rutherford and Westfall 1994).

Environment Of The Grassland Biome

The Grassland Biome encompasses the mountainous region of the south Eastern Cape Province, the high central plateau (Highveld), the Lesotho highlands, the Drakensberg Mountains, and the hills inland of the east coast (Mentis and Huntley 1982; Rutherford and Westfall 1994:32,48). This region is underlain by sandstone, mudstone, and shale formations of the Karoo Sequence formed approximately 330 to 160 million years ago. The

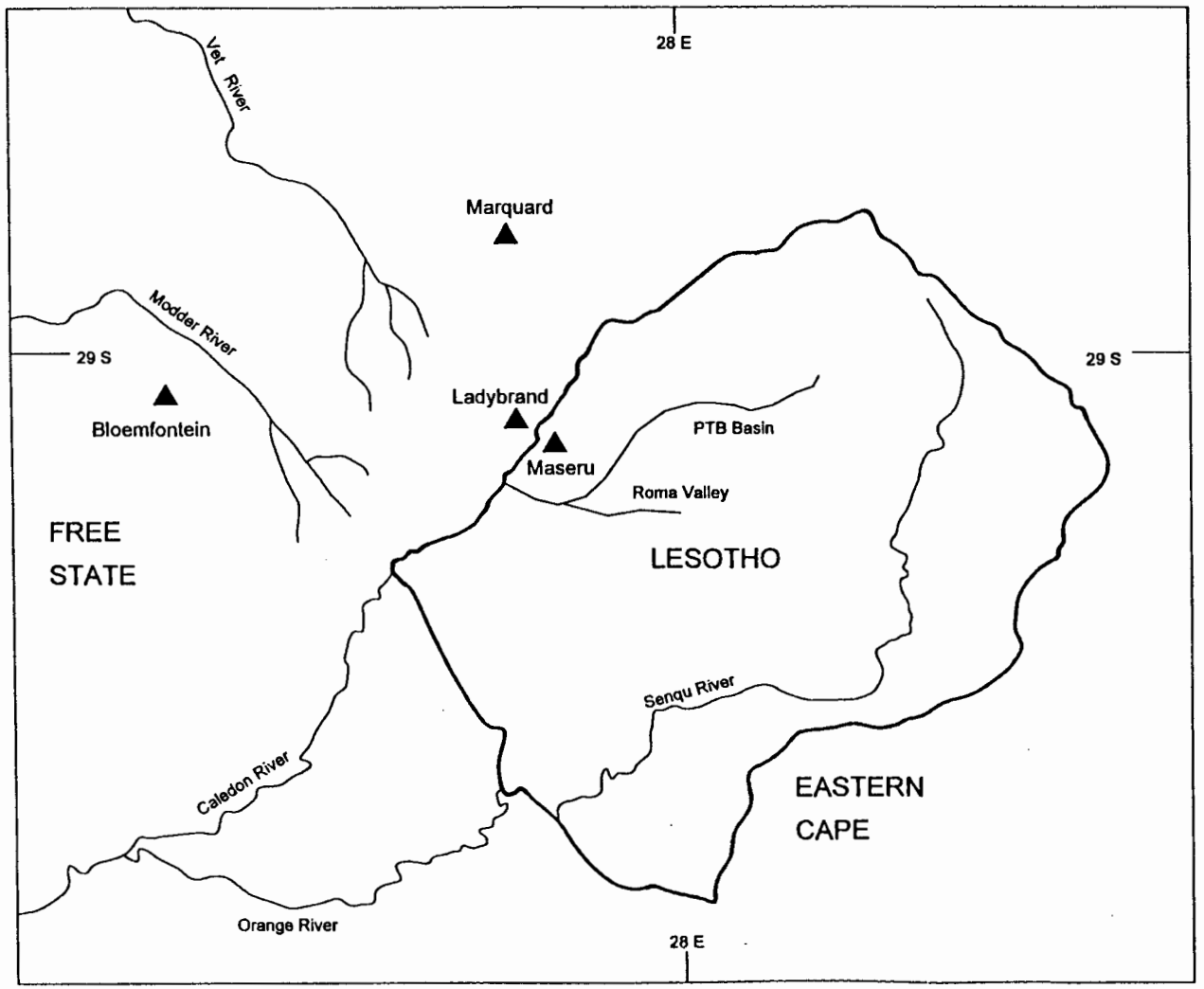

Figure 4.1 Location of Ladybrand, Marquard, Roma Valley, and Phuthiatsana-ea-Thaba Bosiu (**PTB**)

Karoo Sequence is interrupted by outcrops of prior volcanic and sedimentary deposits, as well as extrusive basalt and intrusive dolerite from subsequent volcanic activity (Truswell 1977). Geological processes acting on these deposits formed a region that varies in altitude from 300m to over 3300m asl.

Climate

Growth and distribution of vegetational types within and between biomes are primarily influenced by temperature and the precipitation available for use by plants (Ellery *et al*. 1991; Rutherford and Westfall 1994:4). Often the range for the mean annual temperature has been used as a criterion by which vegetation and biomes have been classified. This has proven to be an inadequate definition, for the mean annual temperature of the Grassland Biome ranges from 16 to 22°C, which is comparable to the rest of the subcontinent (Tainton 1984:12-14, 16-17; Ellery 1992:41). Therefore, Rutherford and Westfall (1994:6) suggest that temperature extremes between the hottest and coldest months provide a more reliable indicator of vegetation distribution. For the Grassland Biome the mean daily maximum temperature for the hottest month, January, is between 25 and 30°C, and the mean daily minimum temperature for the coldest month, July, is near 0°C (Ellery 1992:41; Rutherford and Westfall 1994:48).

Rainfall within this biome is highly seasonal, falling in the summer months between October and April (Rutherford and Westfall 1994:48). The rains begin to fall in the east and move west with decreasing intensity as the season progresses. There is a gradient in the mean annual rainfall from approximately 400mm in the west to 2000mm in the east (Tainton 1984:12-14, 16-17; van Oudtshoorn 1992:41). Plant growth is most productive in early and late summer, but diminishes in mid-summer when heavy downpours accompanied by increased temperatures and light intensity, and higher rates of runoff, evaporation, and plant transpiration limit water availability in soils and lower CO_2 concentrations (Tainton 1984:17). A limited amount of winter precipitation does fall, but low temperatures and frequent frost preclude plant growth.

Soils

Temperature and precipitation are the primary climatic factors influencing soil formation. By influencing both soil moisture and temperature, these two climatic variables in turn affect weathering of soil minerals and the translocation of soil constituents within the profile through: leaching, decomposition of organic matter, and activities of soil fauna and flora (Schmitz and Rooyani 1987:138). Jenny (1941) suggests that soil formation is the result of a combination of factors,

with the impact of temperature and precipitation being enhanced or diminished by the parent rock material, topography, and time.

In areas of high rainfall extremely weathered soils with poorly developed horizons occur. Due to extreme leaching these ferrallitic soils become dystrophic, and consequently have little nutrient value. This impacts directly on the vegetation they support. Soils associated with regions of low to intermediate rainfall produce less weathered soils. These fersiallitic, solonetzic, and planosolic soils display well developed A and B horizons, and tend to be more eutrophic due to a lower occurrence of leaching and weathering. Such soils are richer in nutrients.

Soils developed on intrusive dolerite and extrusive volcanic rock tend to erode more rapidly, once exposed, than the surrounding sedimentary rocks. A eutrophic montmorillonitic clay is produced that is high in nutrients and sustains vegetation that differs from the surrounding soils.

In mountainous regions shallow soils persist as a result of low temperatures that limit weathering, and erosion of steep slopes. Theses soils are low in nutrients and sustain limited, but hardy, plant growth.

The Effect Of Climate On Distribution Of Vegetation

Although there are local variations in temperature, precipitation, and soil, overall these conditions favour a vegetation community that is 75 to 95% C_4 grasses (Vogel *et al.* 1978:214), primarily species of *Cymbopogon* (Turpentine) and *Themeda triandra* (Rooigras) (Acocks 1988:100-112; Rutherford and Westfall 1994:49). The remaining percentage is made up by woody scrub vegetation. Above 2000m asl the percentage of C_4 grasses begin to diminishes with increased altitude to between 25 and 75%, and temperate C_3 grasses become more common, due to a decrease in both evaporation and the mean maximum and minimum growth season temperatures (Killick 1978; Vogel *et al.* 1978:212; Tainton 1984:14; Acocks 1988).

Climatic models for the transitional period from the terminal Pleistocene to the Holocene in southern Africa propose that environmental changes are closely tied to shifts in the seasonality of rainfall, due to deviations in the pattern of atmospheric circulation (Tyson 1986; Cockcroft *et al.* 1987; Tyson and Lindsay 1992). However, it is clear that when considering the Grassland Biome, past changes in the composition of vegetational communities may also be associated with variations in temperature within a normal rainfall season. For this reason both temperature and rainfall are factors that

need to be considered when interpreting isotopic data.

In low-altitude grassland areas where the mean maximum temperatures during the growth season are above 28 to 30°C, seasonality of rainfall is considered to be the most likely cause. Isotopic studies, similar to this one were undertaken at Equus Cave, located in the Grassland/Savanna Biome of the Northern Cape. Given that past temperatures in this area are unlikely to have to dropped below a maximum of 25°C and a minimum of 8°C required to sustain C_4 grasses, researchers eliminated temperature as a primary variable (Lee-Thorp and Beaumont 1990, 1995). They concluded that fluctuations in the C_4 grass component in a level dating between ca. 12 000 - 8000 BP were the result of a normally summer rainfall pattern that extended into the cooler winter months (Lee-Thorp and Beaumont 1990, 1995).

In the high-altitude summer rainfall grasslands of South Africa and Lesotho, temperatures during the growth season become the defining variable, as they are near or below the threshold for C_4 grasses. A lowering of temperature with increased altitude, by approximately 0.6°C for every 100m, creates a transitional area between C_3 and C_4 grasses at ±2300m asl (Table 4.1). A slight

Table 4.1 Estimated altitudinal temperature gradient based on *0.6°C/100m increase between Oxbow Station and Maseru Station

Station	Elevation	Recorded Mean Max/Min January °C Temp	Estimated Mean Max January °C Temp
Oxbow	2600	21.6/2.8	
	2500		22.2
	2400		22.8
Mokhotlong	2300	23.9/9.3	23.4
	2200		24.0
	2100		24.6
	2000		25.2
	1900		25.8
	1800		26.4
	1700		27.0
Ladybrand	1600	27.3/13.3	27.6
Maseru	1500	28.0/15.5	28.2

*(28.0°C [Maseru]/21.6°C [Oxbow])/11 [100 intervals] = 0.58°C/100m

*Based on temperatures data from Killick 1978, Schmitz and Rooyani 1987, and Weather Bureau Reports.

temperature change would result in the expansion of one at the expense of the other. For southern African high-altitude grasslands there is a lack of comparable isotopic data from which to determine whether or not such expansions took place during the terminal Pleistocene or the Holocene. Isotopic evidence that does exist for these higher regions comes from the late Pleistocene. The $^{13}C/^{12}C$ ratios of fossil grazers from Melikane Cave, Lesotho, (Vogel 1983:393) and soil organic matter from the Clarens area, northeastern Free State, (Botha *et al.* 1992:511) indicate that temperatures at this time were sufficiently low to cause an expansion of C_3 grasses.

The sites in the Caledon River Valley do not fall neatly into either of the above mentioned scenarios for the Grassland Biome. A shift in either seasonality of rainfall or temperature could result in vegetational change. In western Lesotho, for example, C_3 grasses are present in the high-altitudes of the Maluti Mountains. The proposed decrease of 3 to 6°C for southern Africa during the Last Glacial Maximum (LGM) (Scott 1986; Heaton *et al.* 1986; Talma and Vogel 1992), sometime between 24 000 and 16 000 years ago, would have brought temperatures down to or below the 25°C/8° C_4 threshold, and allowed for the expansion of the C_3 grasses. On the other hand, present temperatures holding, an extension of the summer rainfall season into the cooler late

summer/early autumn months would increase the percentage of C_3 grasses in the area.

As these factors will influence the interpretation of the data set certain assumption have been made. First, where similarities in data exist between sites in the eastern Free State and the Lesotho sites, it is assumed that seasonality of rainfall and growth temperature were the same. Secondly, where the data differs between these two localities temperature is considered to be the driving force. In this situation, changes in rainfall would affect all the sites, but altitudinal expansion of grasses due to temperature would only be registered in grazers that spent a large portion of the season in the Lesotho grasslands, and not those near the eastern Free State sites. Lastly, it is also assumed that differences between the sites do not reflect micro-environments that promote either C_3 or C_4 grasses, due to soil fertility and moisture, or slope direction. Soil fertility and moisture for both Ladybrand and the Phuthiatsana-ea-Thaba Bosiu River Basin are similar and favour C_4 grasses (see Chapter five).

Additionally, it should be noted that the Grassland Biome is not an isolated vegetational unit and may contain elements of adjacent biomes (Nama-Karoo and Savanna), as plants have a range of tolerance (Tainton 1984:9); for example, the establishment of patches of

woody C_3 vegetation within the climax grassland. Geological, soil, and topographic influences are possible modifiers contributing to the presence of trees and shrubs. In an environment that is normally too dry and has too many frost days to support woody vegetation, gullies and dolerite or sandstone outcrops offer protection against frost, fire, and grazing, and retain sufficient moisture to sustain such vegetation growth (Guillarmod 1971:17; Acocks 1988:100). It has also been debated that climax grasslands occur on soils that have well developed illuvial B-horizon, and where poorly developed woody vegetation will grow (for discussion see Ellery 1992). Although several plant types can survive in an environment, eventually the one best suited to the available, or changing, factors will dominate (Tainton 1984:9).

In the climatic climax or "pure" grassland region of the Grassland Biome (Veld Type 48-60, Acocks 1988), normal plant succession is terminated at the grass stage by climatic conditions (Tainton 1984:4; Acocks 1988:100; Ellery *et al.* 1991). For example, low winter temperatures in the Drakensberg Mountains and high aridity over a large portion of the Highveld prevent succession from advancing to the forest stage in these areas (Tainton 1984:5). In other regions fire, resulting from climatic (Tainton and Mentis 1984; Ellery *et al.* 1991:502; Ellery 1992:26) or human (Acocks

1988:3,5,9-11) factors, as well as agricultural activities, grazing, and soil properties (Tainton 1984:5) create and maintain a disclimax or "false" grassland (Veld Types 61-68, Acocks 1988). Due to natural stability, climatic climax grasslands are sustained regardless of these factors (Tainton & Mentis 1984; Ellery *et al*. 1991).

The overall origins and maintenance of the grasslands has been debated. Acocks (1952:22, 26, 1988:3,5,9-11), West (1952:66), and White (1978:510) propose that the Grassland Biome is a relatively recent phenomenon, and the result of anthropogenic intervention. They postulate that since the arrival of Late Iron Age Farmers, in about the 16th century AD (Maggs 1976) there has been an increase in human-induced fires and manufacturing of charcoal, and clearing of trees for plant cultivation and domesticated grazers. These developments led to the denudation of the forest, with replacement by grassland. Subsequent work has questioned this interpretation (Guillarmod 1971:31; Killick 1978:528; Scott 1989:115; Ellery 1992:21-26; Meadows and Linder 1993:352). Based on palaeobotanical, archaeological, geological, biogeographical, and ecological evidence, these researchers suggest that the grassland, both climax and disclimax, is a construct of climate and has been in existence for millennia. Although in place for a considerable time depth, the

grassland composition and the forces invoking and sustaining its existence may have varied through time.

Fauna

Historical records suggest that in the past a wide diversity of ungulates herbivores contributed to the faunal assemblage of the Grassland Biome (du Plessis 1969; Ansell 1972(a)(b); Lynch 1983; Smithers 1992). Those of importance to this study are the following species of grazers: *Connochaetes gnou* (black wildebeest), *Connochaetes taurinus* (blue wildebeest), *Damaliscus dorcas phillipsi* (blesbok), *Alcelaphus buselaphus* (red hartebeest), *Redunca arundinum* (reedbuck), *Redunca fulvorufula* (mountain reedbuck), *Equus quagga burchelli* (burchell's zebra), and *Equus zebra* (mountain zebra). Also included in the study are two species of browsers, *Pelea capreolus* (grey rhebok) and *Oreotragus oreotragus* (klipspringer). Presently these species inhabit the biome in small numbers, having been over exploited during the latter part of 19th century AD. Further deterioration and shrinkage of suitable habitat has been caused by human settlement, horticulture, and overgrazing by domesticated sheep, goat, and cattle (du Plessis 1968; Ansell 1972(a)(b); Lynch 1983). As a result, what is known about the animals' preferred habitat and migration patterns is often based on the records of early travellers and

observation of modern populations under restricted conditions.

The migration patterns of grazing ungulates coincide with the change in seasonal nutrient composition of the grassland. Following the climate gradient and soil distribution of the biome, grasses are divided into sweet, sour, and mixed velds (Fig. 4.2) depending on nutrient value and palatability at various times of the year (Ellery 1992:1; van Oudtshoorn 1992:37). Grasses with high nutrient values relative to carbon and palatable throughout the year will be sweet, whereas sour grasses are only palatable during the growing season (Ellery 1992:139). The ratio of nutrients to carbon is influenced by the degree of light intensity, water availability, and temperature. Increase in light intensity will increase the photosynthetic uptake of carbon. During the growth season low mean daily temperatures and high moisture favour carbon assimilation over nutrient mineralization (Ellery 1992:144). Nutrient-forming microbes are most productive at higher temperatures and when moisture is just below the wilting point of plants. The effect of light intensity and temperature may be modified in situations where soil properties affect the retention or leaching of available water and nutrients (Ellery 1992:144). In certain micro-environments a mixed veld occurs where conditions support the growth of both sweet

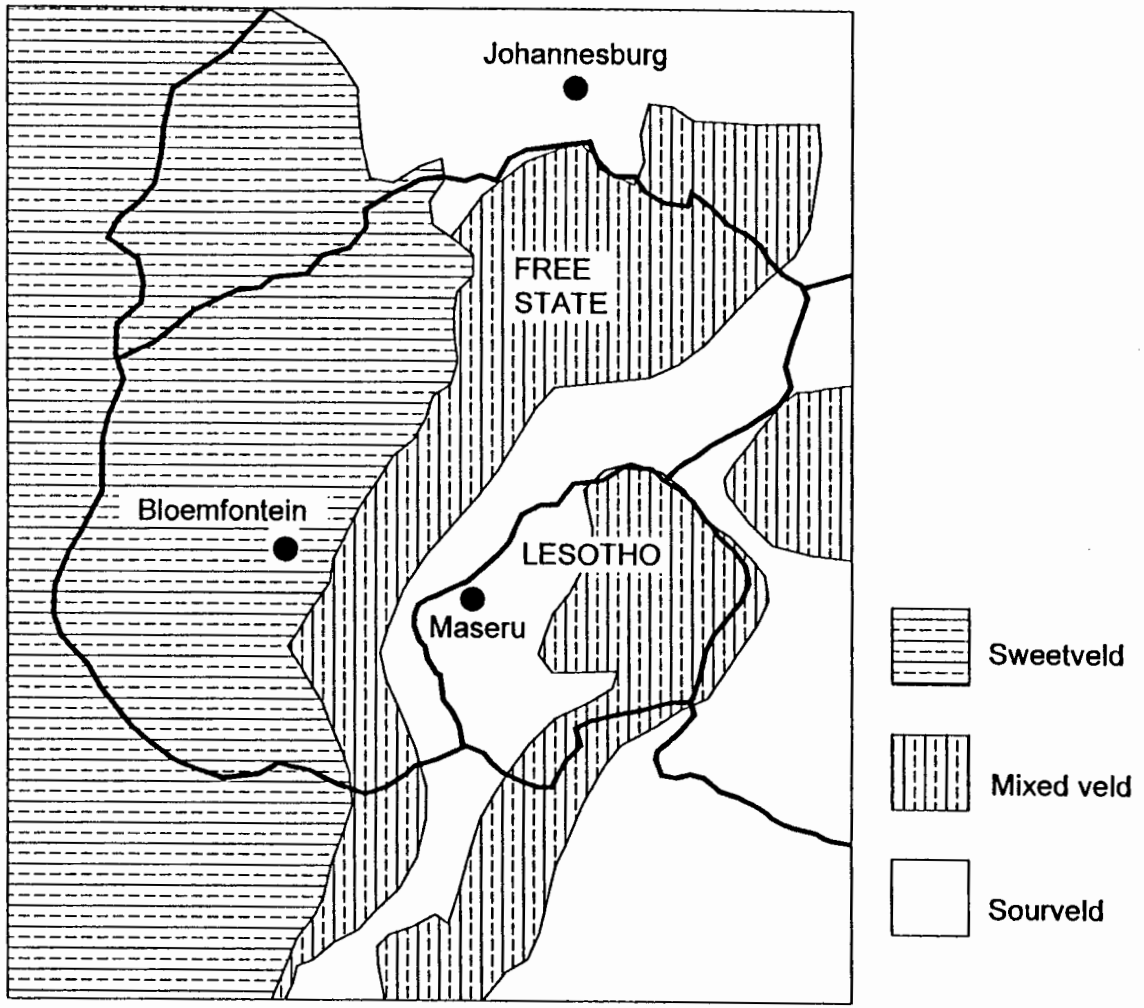

Figure 4.2 Distribution of the veld types within the study area (adapted from van Oudtshoorn 1992)

and sour grasses (Tainton 1984; van Oudtshoorn 1992:39).

Sourvelds are associated with areas where rainfall exceeds 625mm per annum, and mean daily temperatures, around 25°C, are lower than those for sweetvelds (van Oudtshoorn 1992:39). Soils tend to be dystrophic sandstones and quartzites, which retain water and are nutrient poor (Ellery 1992:142-144, 162). Sourvelds are palatable for approximately the first six months of the growing season (Tainton 1984:43). The initial moisture stimulates nutrient assimilation and carbon uptake is limited by a low leaf area (Ellery 1992:170), making the grasses suitable for grazing. In the latter portion of the season, as leaf surface area enlarges and moisture increases, carbon assimilation is continued, while nutrients are leached or no longer produced (Ellery 1992:170). Palatability and nutrient quality of the grasses are diminished and remain so until the beginning of the next growth season. At this point grazers migrate to more acceptable sweet or mixed velds.

Sweetvelds are identified with hot arid areas that have between 250 to 500mm of annual rain, and eutrophic soils comprised of hydrophobic and nutrient rich clays, basalts, and dolerites (Ellery 1992:142-144,162; van Oudtshoorn 1992:37). These conditions produce grasses that are low in carbon, high in nutrients, and palatable year round (Tainton 1981:43; Ellery 1992:170). A lack

of suitable grasses may cause certain species of grazers, in particular *C. gnou* and *D. dorcas*, to browse (Lynch 1983; Smithers 1992). Nevertheless, these periods tend to be sporadic in occurrence, and therefore, unless the situation is prolonged, C_3 vegetation is unlikely to contribute a great deal to the overall $\delta^{13}C$ values of grazers.

Historical sources indicate that grazers migrated from the sourveld of the eastern Free State, Mpumalanga, and Gauteng to the sweet and mixed velds of the western and central Free State, and the KwaZulu-Natal side of the Drakensberg Mountains (du Plessis 1969; von Richter 1971:37-38; Lynch 1983). von Richter (1971:38) suggests that the mixed veld and warmer temperatures of KwaZulu-Natal served as a winter refuge for grazers, and that they migrated westward at the start of summer rains when the sourvelds of the eastern Free State and Gauteng are palatable. In late summer populations migrated to the sweet and mixed velds of central and western Free State.

Summary

The vegetational composition of the Grassland Biome is influenced by temperature, the availability of water, and soil properties. In most areas of the Grassland Biome temperatures are sufficiently high enough that should there be a decrease in temperatures they would

not fall below those required to sustain a purely C_4 grassland. The exception to this is the high-altitude summer rainfall regions of Lesotho, where a decrease of 0.6°C would result in the expansion of C_3 grasses. When reconstructing past environments the stable carbon isotopes of soil organic matter will reflect *in situ* vegetational growth, providing no disturbance has occurred to the sedimentary sequence. This chapter has also highlighted the point that the fauna studied will reflect the more regional environment of the Grassland Biome, due to their migratory nature. Thus, it should be realized that the results from faunal material retrieved from sites in the Caledon River Valley may represent a compilation of a larger regional picture.

CHAPTER 5

SITES AND SAMPLES

Faunal material recovered from the Late Pleistocene and Holocene archaeological deposits at the sites of Rose Cottage Cave and Twyfelpoort Shelter, in the eastern Free State, and the rock shelters of Tloutle, Ha Makotoko, and Ntloana Tsoana, in western Lesotho (Fig. 5.1), were sampled for stable carbon isotopic analysis. Grasses and soil organic matter from the Rose Cottage Cave area were also sampled for corresponding isotopic analysis.

Because the acidic cave sediments have caused poor preservation of bone and bone collagen, enamel was the main source of material sampled. When present in sufficient quantity and quality, bone collagen was analysed in conjunction with enamel apatite as a means of verifying results. In order to maximize information and guard against possible anomalies, more than one species, as well as more than one individual of each species were analysed from each stratum. However, sufficient material was not always available to allow for this selection process.

In the sampling strategy, consideration has been given to: 1) the biological requirements of the species of animals or plants; 2) the spatial and temporal context

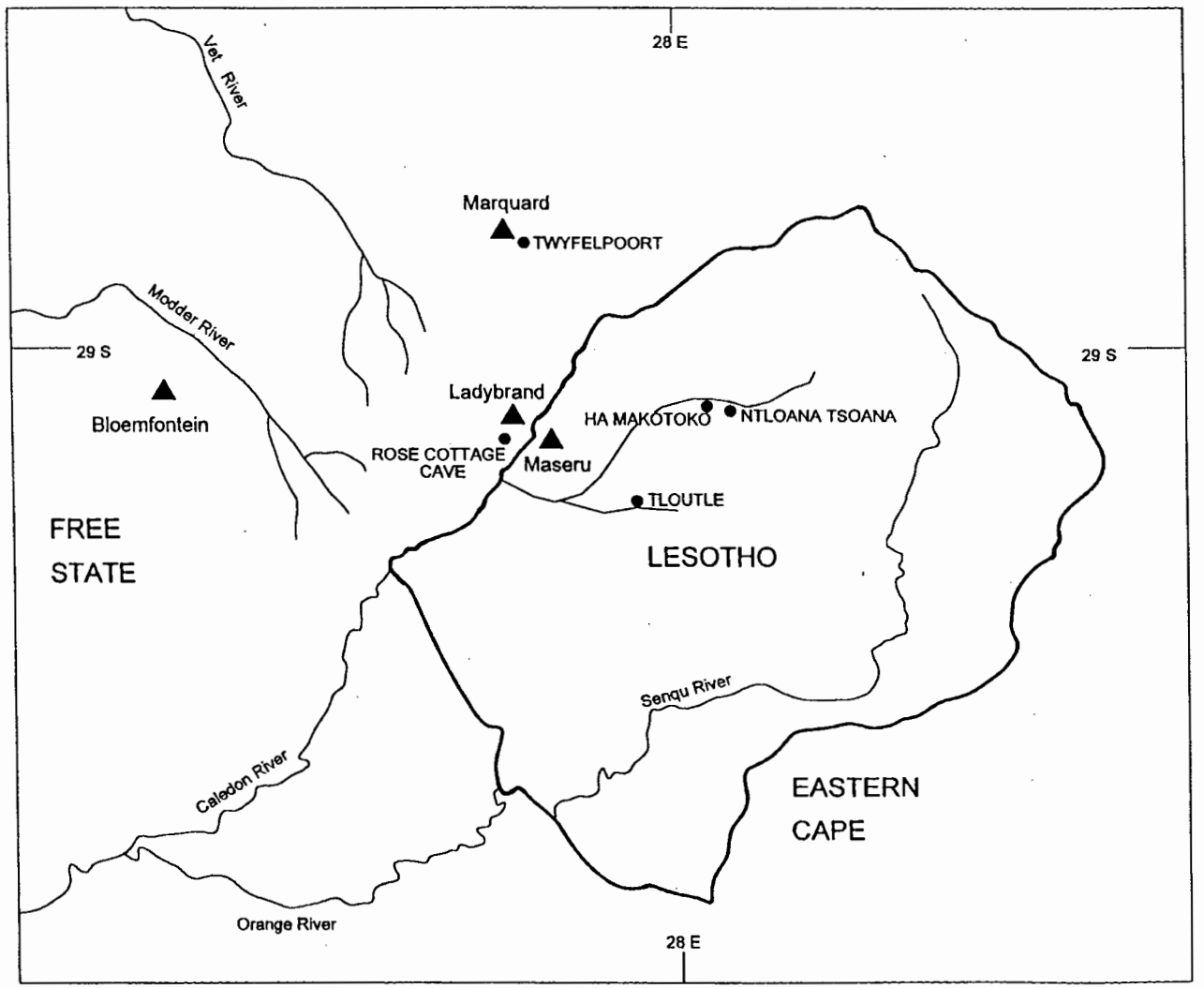

Figure 5.1 Location of Rose Cottage Cave, Twyfelpoort, Tloutle, Ha Makotoko, and Ntloana Tsoana

of fauna and vegetation prior and subsequent to deposition, and 3) state of preservation after deposition. Furthermore, attention has been given to other palaeoenvironmental evidence, such as the study of charcoal (Esterhuysen 1996; Esterhuysen and Mitchell in press); macro-fauna (Plug and Engela 1992; Plug 1993), and sediment deposition (Butzer 1983, 1984(a)(b)). These sources provide evidence for past environmental conditions that complement the stable carbon isotopic analysis.

Rose Cottage Cave (RCC)

Local Environment

Rose Cottage Cave is situated on the Platberg, approximately 5km east of the town of Ladybrand (29°13'S;27°28'E) in the eastern Free State, and about 10km north-west of Maseru in western Lesotho. The terrain is between 1500 and 1800m asl, and is characterized by flat gently rolling plains and outcrops of sandstone and mudstone. The Platberg is derived from the Clarens Sandstone Formation of the Karoo Sequence, with the cave located on the northern slope at an altitude of ±1660m asl. Cave formation took place through differential erosion of the sandstone, and was filled through several thousand years of aeolian and alluvial deposition, and weathering of the interior cave

surface (Butzer 1984a), and cultural occupation (Wadley 1991, 1995, 1996).

For Ladybrand the recorded mean daily maximum and minimum temperatures for the hottest month, January, are 27.3°C and 13.3°C, and for the coldest month, July, they are 15.4°C and 0.1°C respectively (Weather Bureau 1911-1940). Although the rainy season for Ladybrand begins in October, most of the precipitation falls between January and April. Mean annual precipitation is 730mm (Weather Bureau 1911-1988). The climate of the Ladybrand area favours a vegetation that is primarily a climatic climax *Cymbopogon-Themeda triandra* grassland of mixed-sourveld quality (Veld Type 48, Acocks 1988). As a part of this research project a local survey of the Ladybrand area was conducted in November 1994 in which the grass species of *Eragrostis sp.* (love grass), and *Aristida congesta sp. congesta* (tassel three-awn) were noted to also be in abundance. Except for *T. triandra*, all of these species are low in both nutritive and grazing quality, and are often indicators of overgrazing and poor grassland management (van Oudtshoorn 1992). A dense woody and scrub-thicket vegetation (Veld Type 56, Acocks 1988) is supported on the slopes of sedimentary outcrops. Although this mixed-veld is dominated by sour grasses, sweet grasses flourish in periods of drought, and *T. triandra* and *A. congesta sp. congesta* stay palatable for longer. Grazing ungulates would have

favoured the Ladybrand area from early to mid-summer and during extended drier intervals.

Site Background

Over the past 50 years Rose Cottage Cave has been the focus of research and excavations. Berry Malan undertook the initial excavation of the cave in the 1940s, in which he excavated a portion of the east section (Wadley 1991). A subsequent excavation of the east section by Peter Beaumont in 1962 yielded radiocarbon dates, which established the antiquity of Rose Cottage Cave's cultural sequence to be greater than 50 000 BP (Wadley 1991; Wadley and Vogel 1991). Thus, the site is only one of a few found in the interior of South Africa to encompass the Middle and Later Stone Ages. With the exception of Karl Butzer's (1984(a)(b)) study on the geological and stratigraphic formations of the site, no further research was carried out at Rose Cottage Cave until the late 1980s.

In 1987 excavation was resumed, this time on the west side of the cave (Fig. 5.2) by Lyn Wadley, Department of Archaeology, University of the Witwatersrand. This section has been excavated according to stratigraphy (Fig. 5.3), to a depth of 0.4m (level Pt) over an area of 38m^2 (Wadley 1995). A further 26m^2 have been

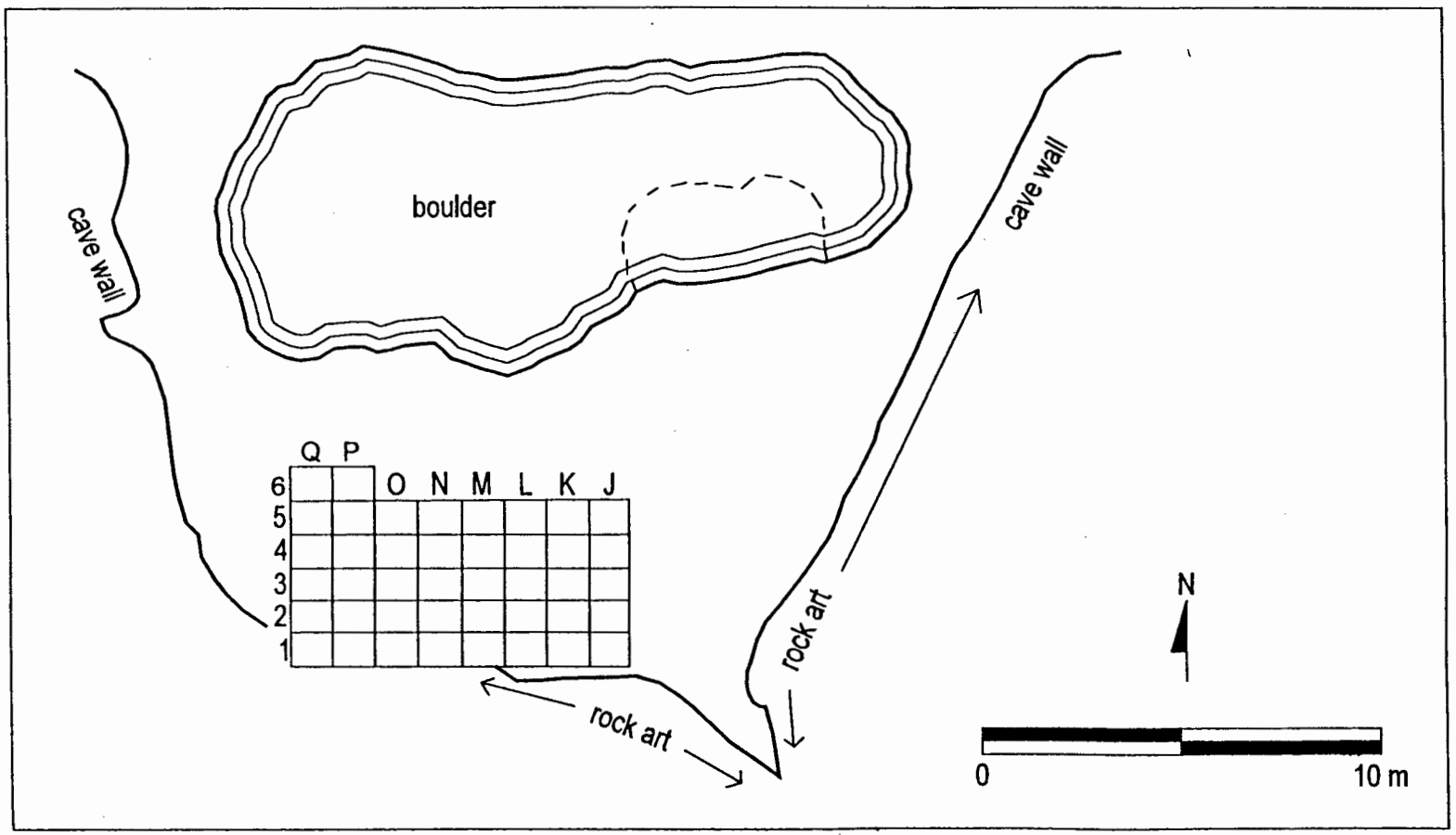

Figure 5.2 Rose Cottage Cave site plan and location of excavation (redrawn from Wadley 1991)

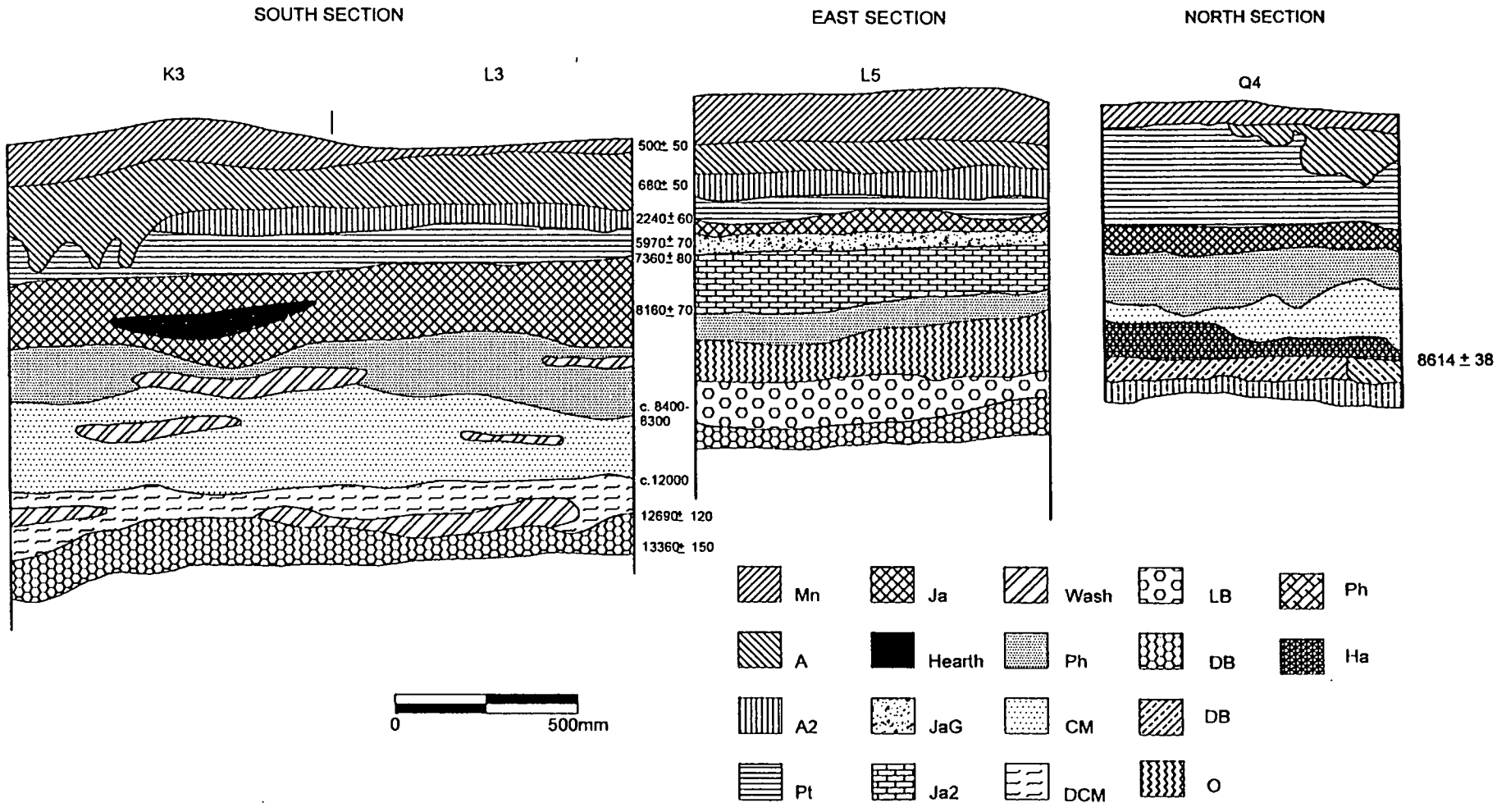

Figure 5.3 Rose Cottage Cave stratigraphic sequence (redrawn from Wadley 1996)

excavated to 1.5m (level G), with a witness trench to 2.3m (Wadley 1991, 1995).

Context Of Faunal Samples

Isotopic analysis was carried out on the bones and teeth of ungulate herbivores recovered from levels of the west section dating between 13 500 and 450 BP. At present, preservation and quantity of faunal material suitable for stable carbon isotopic analysis does not extend beyond level DB.

Levels DB-LR (13 360±150 BP - Pta-5601), DB-UP (12 690±120 BP - Pta-5593), DCM (undated), and LB (9560±70 BP - Pta 7275) contain lithic artefacts attributed to a Robberg-type Industry (Wadley 1996), which is the first fully recognizable cultural occupation following an approximate 7000 year hiatus. DCM and LB were thought to be contemporaneous and date ca. 12 000 BP (Wadley 1996:64), but LB now has a ^{14}C date of 9560±70 BP (Wadley pers. comm.). Relative to the previous and subsequent lithic industries, the Robberg levels contain a large number of conical bladelet cores and bladelets (Wadley 1991:128, 1995:574, 1996:64-66). Worked bone and ostrich eggshell form a small percentage of the assemblage (Wadley 1996:67). Charcoals from level DB and LB indicate that a 'macchia-alpine' type of woody vegetation was in place at this time (Esterhuysen

1996:101). The faunal assemblage is dominated by species of large to medium ungulate grazers (Plug and Engela 1992) that favour grassland or open savanna habitats. Due to poor preservation, no isotopic analysis was conducted on faunal material from level LB, but it may yet be considered for analysis should appropriate material become available. As LB is dated to ca. 9600 BP, material from this level may provide cultural information for the transitional period between the Robberg and the following Oakhurst Industry (Wadley 1996:71), and palaeoenvironmental data for the shift from the terminal Pleistocene to the early Holocene.

Levels O (9250±70 BP - Pta-5599), Ha (8614±38 BP - Pta-5560), Cm (undated), JaG (8380±70 BP - Pta-5600), Ph (8350±70 BP - Pta-7287), and Ja (8160±70 BP - Pta-7117) contain an Oakhurst Industry that is characterized by a macrolithic assemblage containing sandstone and hornfel scrapers and sidestruck flakes (Wadley 1991:128, 1995:574; Engela 1995). Charcoal from these levels is comprised of a high percentage of "woodland" taxa (Esterhuysen 1996:101). The fauna from the Oakhurst level is similar to that of the Robberg, but there is an increase in ungulate browsers and woodland species, such as *Tragelaphus strepsiceros* (kudu), *Cercopithecus aethiops* (vervet monkey), and *Panthera pardus* (leopard) (Plug and Engela 1992; Wadley 1995). Level O lacks preservation of appropriate faunal species, therefore,

no isotopic results are available for this level.

Levels Pt-LR (7630±80 - Pta-6783) and Pt-UP (5970±70 - Pta-5934) are associated with a classic Wilton Industry, in which the lithic component is typified by small scrapers and backed tools (Wadley 1995:574). Woody vegetation similar to that of the Oakhurst period is suggested by the charcoal, but Pt-LR has a larger xerophytic component than Pt-UP (Esterhuysen pers. comm.). Likewise, except for the absence of several woodland species, the faunal assemblage remains comparable to previous levels (Plug and Engela 1992; Wadley 1995).

Levels A2 (2240±60 BP - Pta-7122), A (680±50 BP - Pta-5622), and Mn (500±50 BP - Pta-6788) follow an occupational hiatus and are associated with the post-classic Wilton Industry. The artefact assemblages from these levels contain lithic tools, primarily scrapers and retouched pieces, as well as worked bone and ostrich eggshell, and grass and grit-tempered pottery (Wadley 1992:9, 1995:574). European artefacts have been found in the upper section of level Mn (Behrens 1992). In level A the woody vegetation is dominated by the xerophytic *Acacia karroo*, while in Mn the modern compilation of woody species has been established (Esterhuysen 1996:135-148). The faunal remains indicate that ungulate herbivores are still present; however,

domesticated animals, fish, and amphibian remains are now represented in large quantities (Wadley 1992:9; Plug and Engela 1992).

Context Of Soil Samples

Isotopic analysis was conducted on soil organic matter from sediments from each of these cultural levels. Sediments were sampled from different squares, as one square did not contain all the levels. In a 1979 letter to the National Monuments Council of South Africa, Butzer indicates that, "Malan had sectioned the deposit with a very fine eye as to the critical stratigraphic contacts", thereby enabling him to reconstruct the complex stratigraphy of Rose Cottage Cave, and tie together radiocarbon dates with levels from Malan's excavations. Although not as refined as the west section, Butzer's (1984a:57) proposed sedimentary sequence for the eastern portion of the cave has been extrapolated to form the following sequence for levels Mn to DB (Wadley and Vogel 1991:606):

Mn - A: Brown to dark brown silty sand that is sorted, laminated and crossbedded,

A2: Dark grey to brown, organic silty sand that is poorly sorted with roof spalls,

Hiatus: Sediment break for ca. 3000 years,

Pt - O: Brown to black silty sand that is poorly sorted with roof spalls, and

DCM - DB: Brown to black stony silty sand with large amounts of roof spall.

Soil organic matter was also sampled within the vicinity of the cave from a donga formation (Donga 1) located on top of the Platberg, and from a unit (Unit 1) at the base of hill below Rose Cottage Cave (see Fig. 5.4). Unit 1 is located on the margin between the scrub-thicket of the hill slope and the grassland of the plain. Any shift in either of these communities should be reflected in the soil organic matter. The unit was dug to a depth of 0.60m, at which point the ground was too hard to proceed further. No stratigraphy was visible, therefore, sediments were sampled at 50mm intervals in order to obtain a high resolution. Leaf litter and grasses were collected from near Unit 1 for the purpose of establishing the $\delta^{13}C$ signature of the modern vegetation and decomposing organic matter.

In order to document any changes in the current pure grassland environment over time, it was decided to analyse sediments from the already exposed stratigraphic profile of Donga 1. The donga was sampled from the surface at 0.10m intervals to a depth of 1.00m. Organic material from a depth of 0.70m yielded a radiocarbon date of 1590±70 BP (Pta-6843), with the top 0.20m of the donga representing post AD 1954 (Pta-6839) organic accumulations. These dates are based on the average age of the organic carbon accumulated within the donga during soil formation.

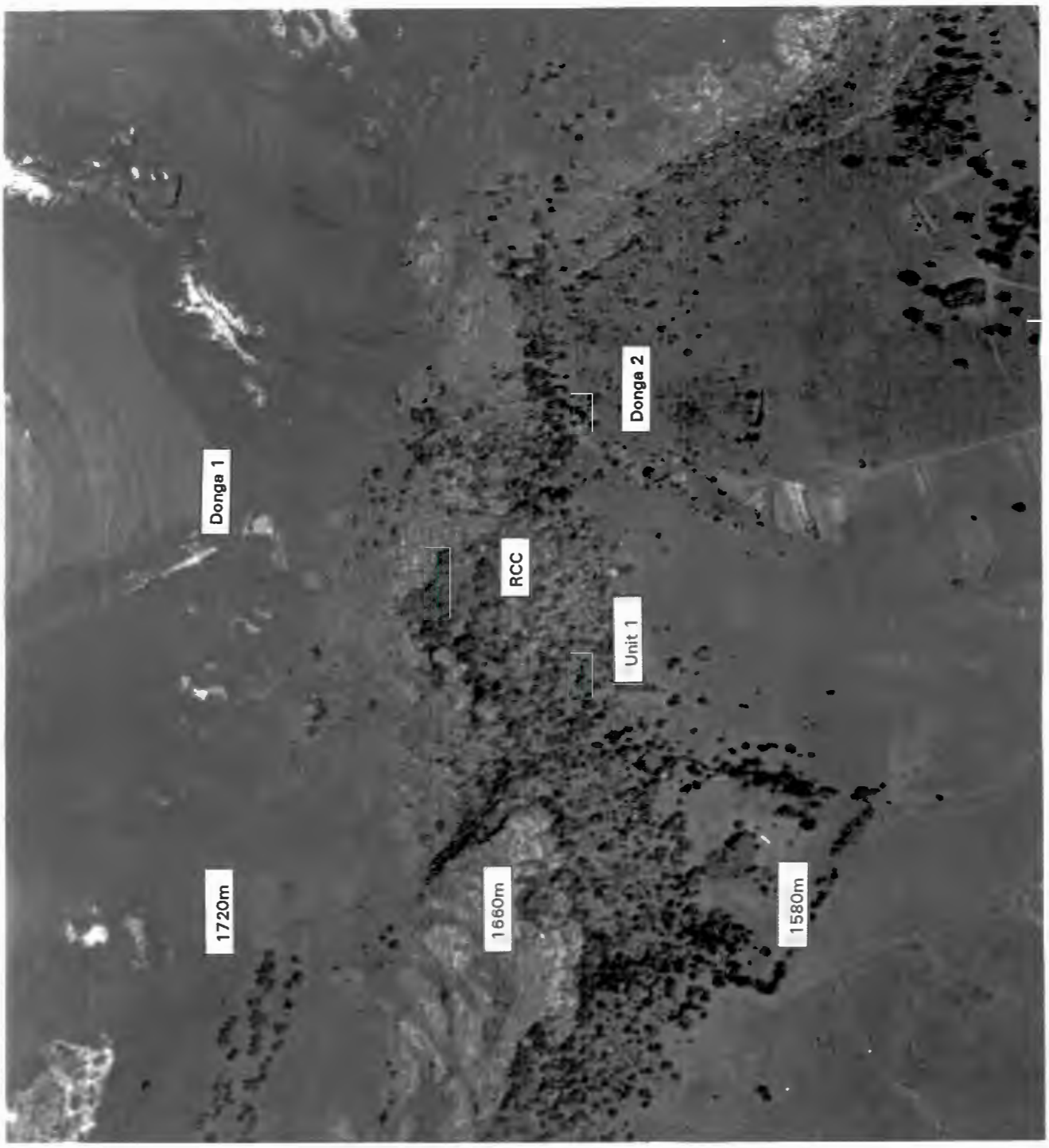

Figure 5.4 Location of sampled soils and sediments

In previous palaeoenvironmental research, Butzer (1983, 1984b) studied the donga (for the purpose of discussion this is referred to as Donga 2) eroding out of an "alluvial fan" located below the Platberg, approximately 100m west of Unit 1 (see Fig. 5.4). He concluded that the upper 1.30m of Donga 2 dates to the last 1100 years, with the upper 0.30m forming the recent humic A-horizon. Below 1.30m is a dark loam horizon, 0.80 to 1.50m thick, that is characteristic of a 'wet micro-habitat' with abundant vegetation (Butzer 1983, 1984b). Organic material recovered from this horizon was dated to ca. 2310 BP (Butzer 1984b:245; Vogel pers. comm). The dark loam soil extends to the upland area of the Platberg (Butzer 1983) and is consider to be associated with the soil from Donga 1 dated to 1590±70 BP (Vogel pers. comm.).

Twyfelpoort (TFP)

Local Environment

Twyfelpoort is located 20km north-east of the town of Marquard (28°37'S;27°34'E). This area has a flat to gently rolling terrain that is approximately 1500m asl, and is underlain by the Clarens Sandstone Formation. Although there are no recorded air temperatures for Marquard, it can reasonably be assumed that they are seasonally similar to that of Ladybrand, approximately

100km to the southeast. The mean annual rainfall is 618mm (Weather Bureau 1916-1950), with the majority of the rain falling between October and April.

The site of Twyfelpoort is situated within a sandstone rock shelter positioned on the south facing slope of a shallow valley. A dense growth of scrub-thicket and trees (Veld Type 58, Acocks 1988) fronts the site, as well as other rocky outcrops and south facing slopes in the Marquard area. A *Cymbopogon-Themeda triandra* grassland of mixed-sourveld quality dominates the landscape (Veld Type 48, Acocks 1988). This grassland would probably have attracted grazing ungulates during the early to mid-summer.

Site Background

Twyfelpoort was excavated by Carol Wallace (1993) as part of an Archaeology Honours project at the University of the Witwatersrand. The eastern section of the rock shelter was excavated to a depth of 500mm over six square metres (Fig. 5.5) (Wallace 1993; Wadley 1995). Ten stratigraphic units were identified (Fig. 5.6) (Wallace 1993). Based on the presence of grit-tempered pottery, glass beads, metal, and peach pips, it is believed that the upper three levels date to within the last 400 years (Wallace 1993). Immediately below these levels a radiocarbon date of 1880±50 BP (Pta-6171) was

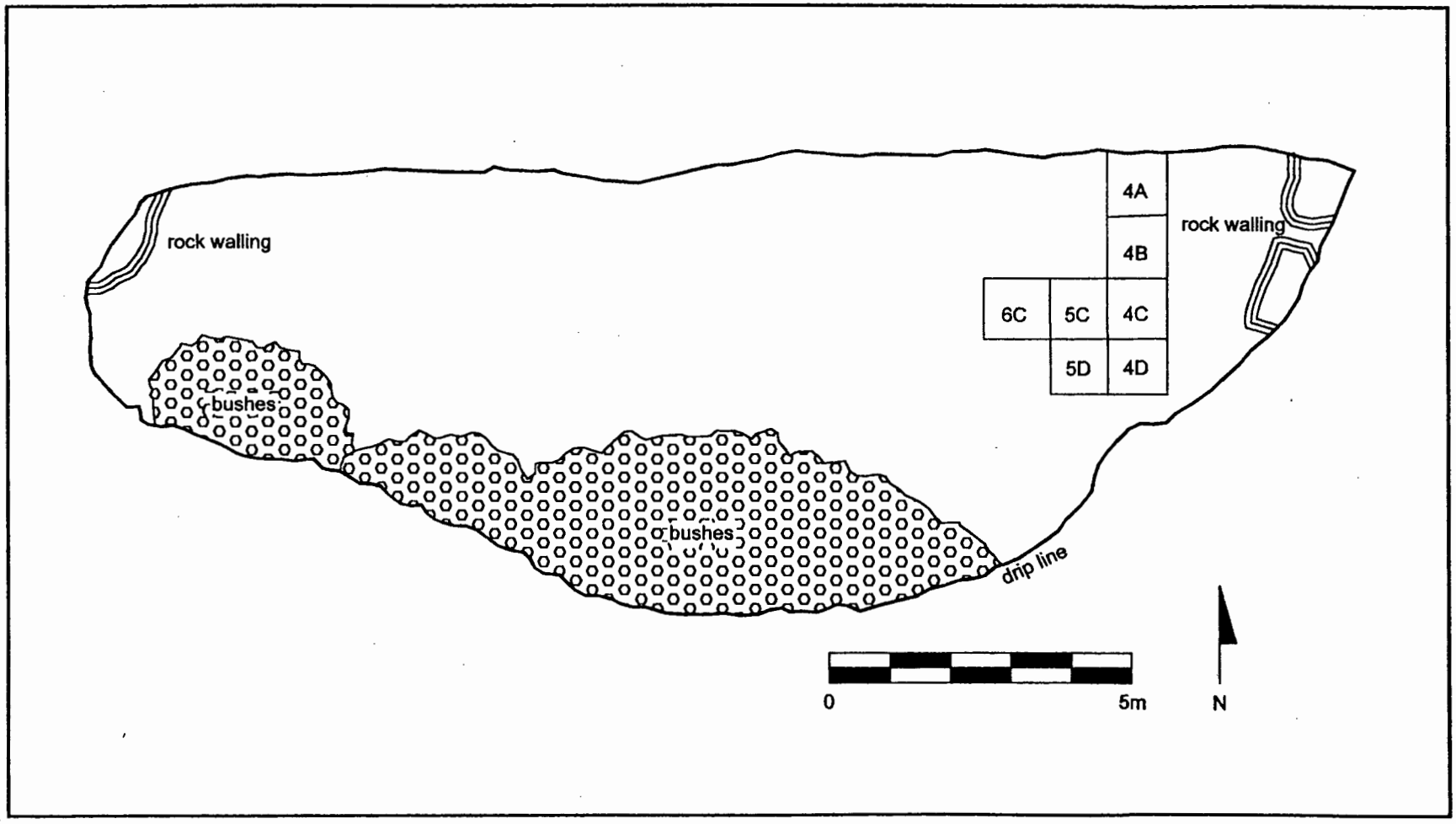

Figure 5.5 Twyfelpoort site plan and location of excavation (redrawn from Wallace 1993)

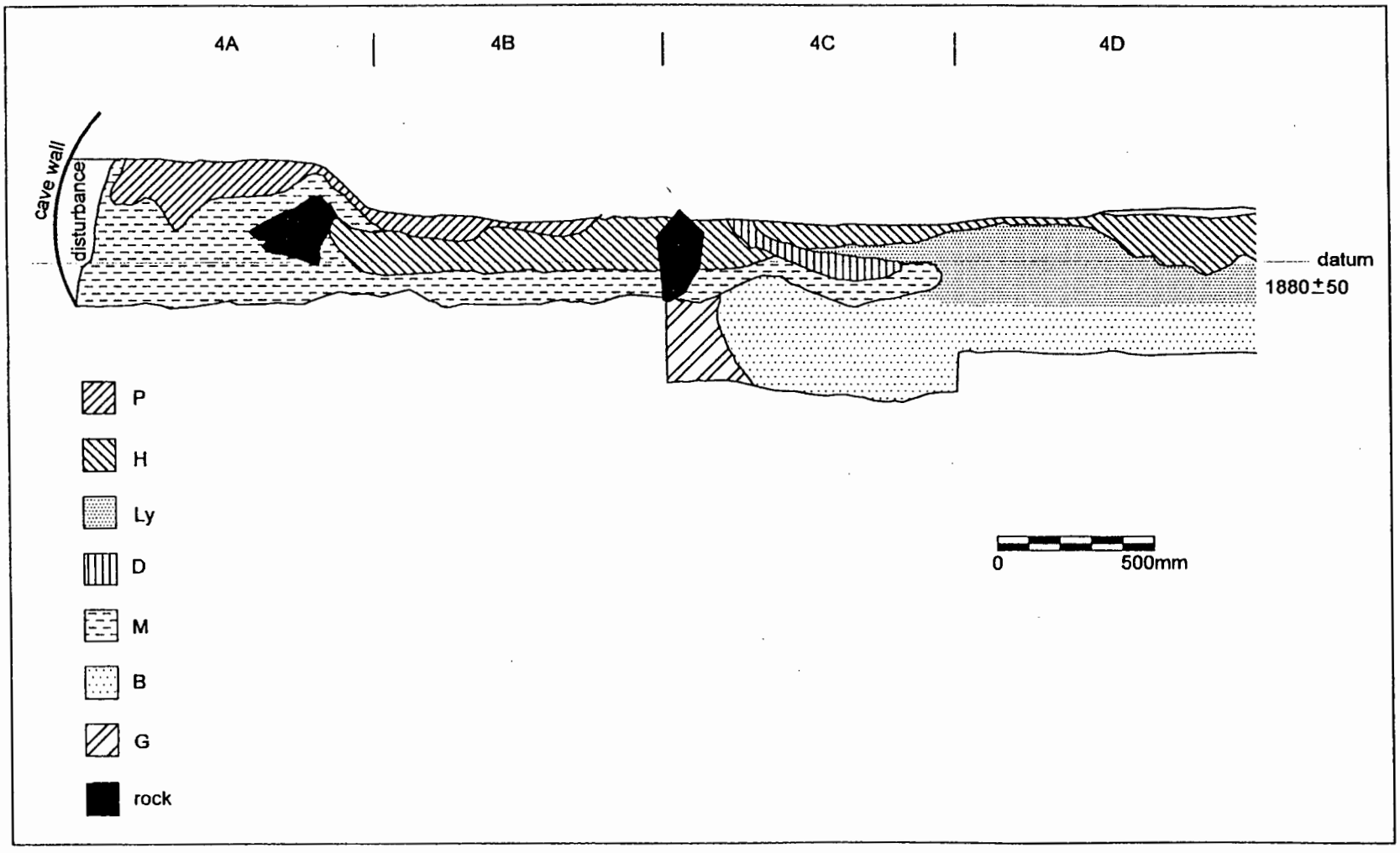

Figure 5.6 Twyfelpoort stratigraphic sequence (redrawn from Wallace 1993)

obtained from the pre-ceramic level Ly (Wallace 1993; Wadley 1995). Below level Ly the chronological order of the remaining stratigraphic units is not clearly defined.

Context Of Faunal Samples

As a result of the lack of stratigraphic definition and poor organic preservation, isotopic analysis was limited to faunal material from level Ly (1880±50 BP). This level appears to represent the last pre-ceramic occupation of the site prior to a 1400 year hiatus. The artefact assemblage is dominated by small scrapers, backed tools, and adzes, along with ostrich eggshell beads and worked bone (Wallace 1993). Charcoal analysis reveals a woody vegetation that is lower in diversity than in previous levels, and is predominately xerophytic scrub (Esterhuysen 1996:16). A large percentage of the identifiable faunal material consists of medium to large sized grazing and browsing ungulates, as well as small mammals, reptiles, and amphibians (Wallace 1993).

Tloutle (TL)

Local Environment

Tloutle (29°28'S;27°46'E) is a sandstone rock shelter located within the Roma Valley. The valley forms a part

of the larger Phuthiatsana-ea-Thaba Bosiu (PTB) River Basin, a tributary of the Caledon River. At an elevation of 1870m asl, the site lies within the region characterized by Bawden and Carroll (1968) and Guillarmod (1971) as the Foothills of Lesotho, and is a transitional environment between the Lowland and the Mountain environments (Fig. 5.7). The site is situated on the west facing slope of an escarpment that marks the boundary between the sandstones of the Clarens Formation and the basalt lavas of the Lesotho Formation (Mitchell 1993a:79, 82).

In general, the climate of the area is temperate and sub-humid (Chakela 1981:19). No direct climatic data is available for the Foothills, but there are fairly detailed records for Maseru (1530m asl), located in the Lowlands of the Roma Valley. Here the mean daily maximum and minimum temperatures for the hottest month, January, are 28.0°C and 14.3°C, while for the coldest month, July, they are 15.5°C and -0.1°C respectively (Mitchell 1993a:39; Chakela 1981:37). Temperatures decrease along an altitudinal gradient from the Lowlands to the Mountains. At Mokhotlong, 2300m asl, maximum and minimum temperatures for January are 23.9°C and 9.3°C, and at Oxbow, 2600m asl, they are 21.6°C and 2.8°C (Bawden and Carroll 1968; Killick 1978). Following the increase in altitude from west to east, the mean annual rainfall ranges from 682mm at Maseru to over 1400m in

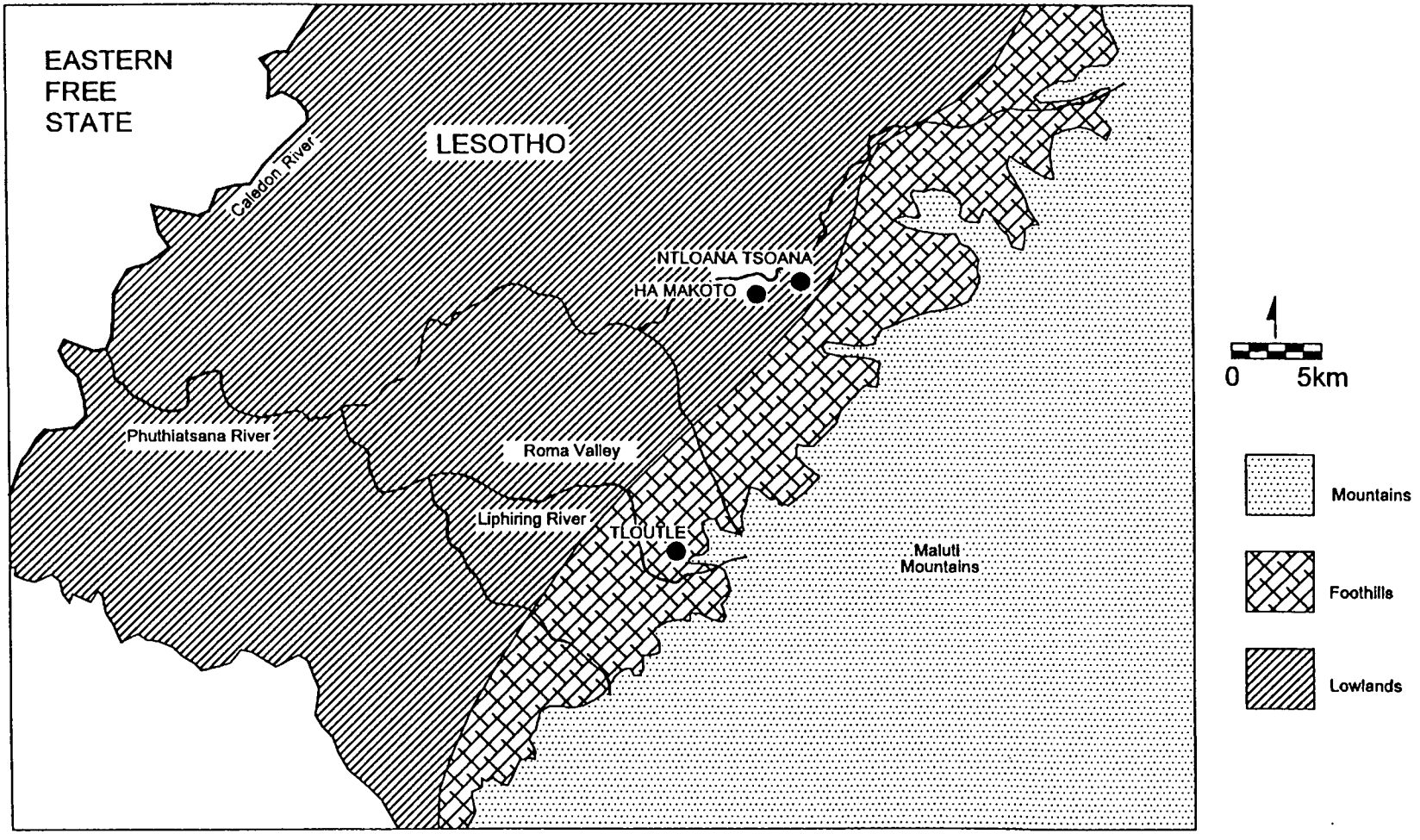

Figure 5.7 Lesotho sites in relation to the Lowlands, Foothills, and Mountains (redrawn from Mitchell 1990)

the Mountain region (Schmitz and Rooyani 1987:139). At Roma, near Tloutle, it is 810mm (Bawden and Carroll 1968). More than 80% of the annual precipitation falls between the warm months of October and April (Chakela 1981:36). In low lying areas winters tend to be cold and dry, with higher altitudes receiving precipitation in the form of snow. Although there is moisture, the sub-zero temperatures prevent vegetation growth in these alpine areas.

The natural grassland of the Foothills reflects a transitional position between the *Cymbopogon-Themeda-Eragrostis* (Veld Types 48, Acocks 1988) of the Lowlands and the *Themeda-Festuca alpine* (Veld Type 58, Acocks 1988) of the Mountains. From this a grassland of mixed-sourveld quality is formed. Due to the extensive over-cultivation of the land, grasses are of poor quality and xerophytic Karoo plants have begun to replace trees and shrubs (Arbousset and Daumas 1968), which are now restricted mainly to gullies, rocky outcrops, and south facing slopes (Guillarmod 1971; Mitchell 1990).

In the past, both grazing and browsing ungulates were present in large numbers (Arbousset and Daumas 1968). Excavated faunal remains include open grassland species such as *E. burchelli* and *C. gnou*, as well as those adapted to hilly slopes such as *R. arundinum*, and *O. oreotragus* (Plug 1993; Mitchell 1994:86). Grasses would

be most appealing to grazers during the early summer.

Site Background

Excavations were carried out at Tloutle in 1988 and 1989 by Peter Mitchell, Oxford University (Mitchell 1990, 1993a). Two areas of the site, designated as "exterior" and interior", were excavated according to stratigraphic units down to bedrock (Fig. 5.8). Eight square meters were removed from the exterior excavation situated in the northern part of the shelter, from which eighty-two defined stratigraphic units were grouped into nine layers. The lower six layers of the excavation (Fig. 5.9) produced an early to middle Holocene archaeological sequence, with dates ranging from ca. 8700 - 6100 BP (Mitchell 1990, 1993a). The top three layers, SS, BGL and WC, may belong to the more recent post-classic phase of the interior Wilton Industry (Mitchell 1993a:121). Due to a lack of both suitable faunal preservation and stratigraphic integrity, the faunal material from the upper levels was not analysed in this study.

The interior excavation located near the back wall of the rock shelter revealed a heavily disturbed deposit, resulting from the recent usage of the rock shelter by the local Basotho people and their livestock (Mitchell 1993a:84). For this reason the isotopic data from this

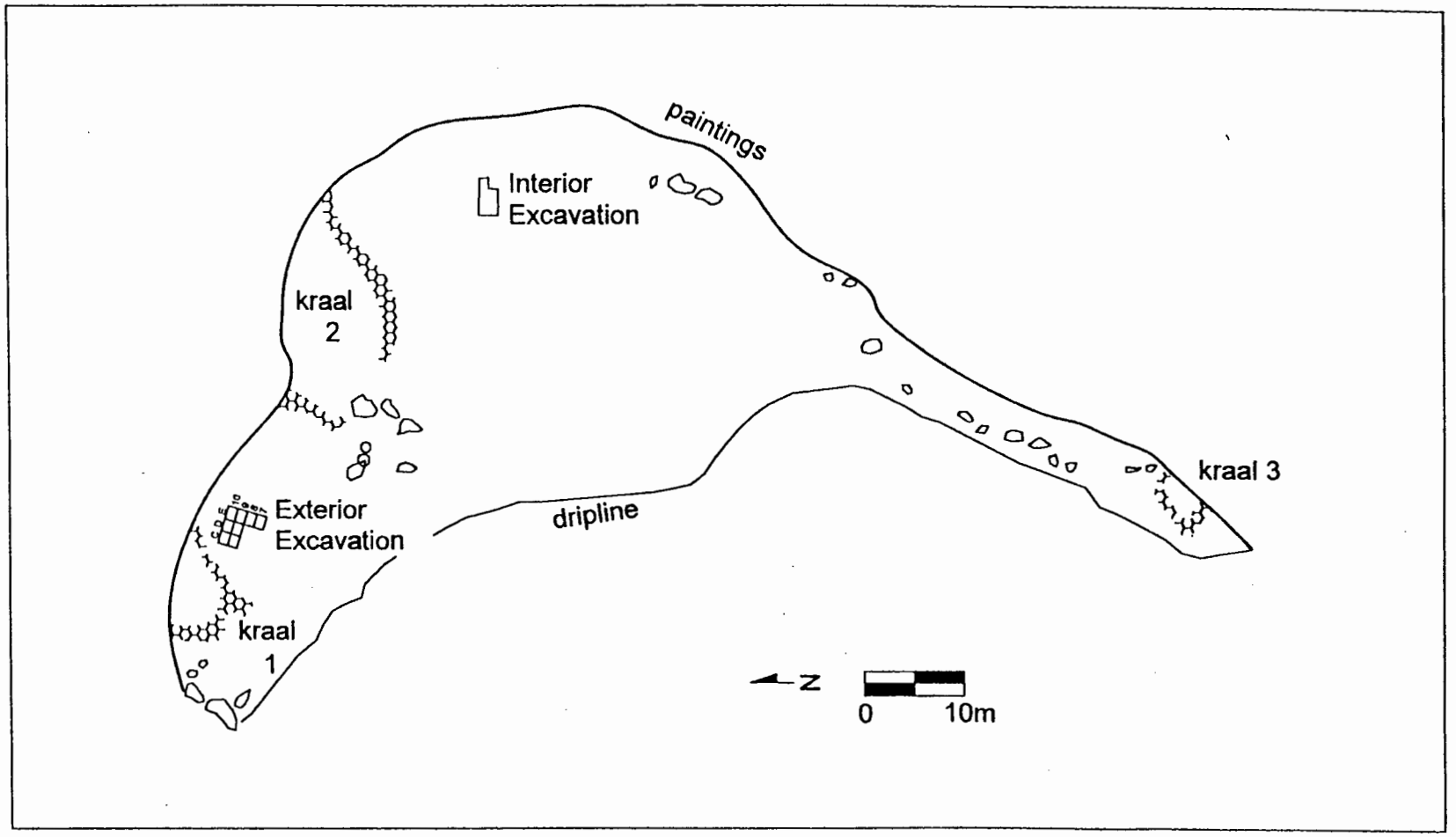

Figure 5.8 Tloutle site plan and location of excavation (redrawn from Mitchell 1990)

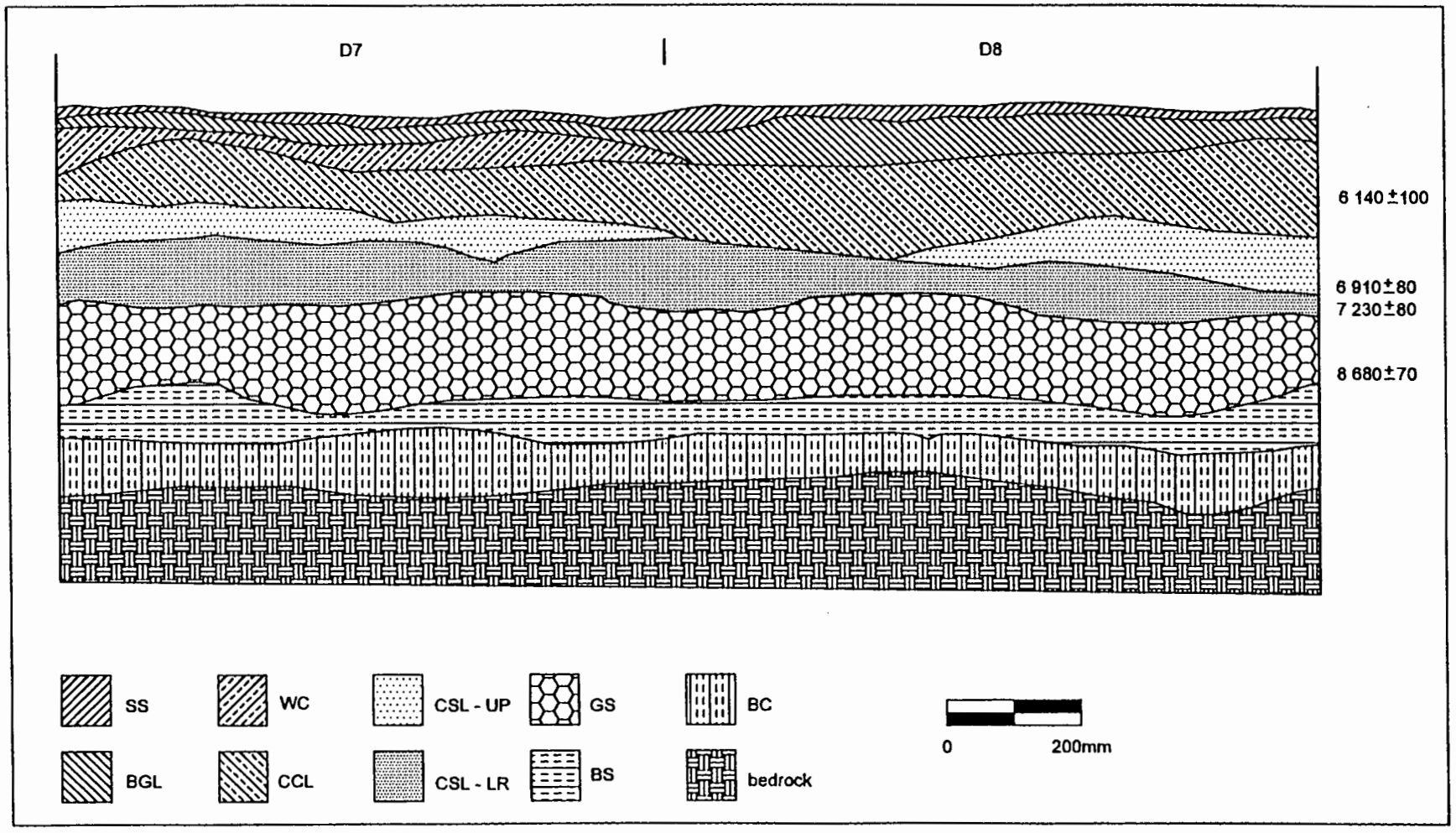

Figure 5.9 Tloutle stratigraphic sequence
(redrawn from Mitchell 1990)

excavation are given in Appendix I section C, but not discussed in this thesis.

Context Of Faunal Samples

Layers BC (undated) and BS (undated) occur near the base of the excavation and contain lithic assemblages with high-backed bladelet cores. Mitchell (1990:102, 1993a:88) suggests that layers BC and BS are probably associated with a Robberg-type Industry dating ca. 13 500 - 12 000 BP, although BS may range from ca. 9500 - 12 000 BP (Mitchell pers. comm.). Faunal material from these levels is limited, and that which is recorded consists of medium to large bovids, with small to medium mammals also present in layer BS (Plug 1993). Associated botanical information is unavailable for layers BC and BS, due to a lack of charcoal perseveration (Esterhuysen 1996:72). Samples from three individual grazers, two from BC and one from BS, were analysed in this study.

Layer GS (8680±70 BP - Pta-5172) is associated with the 'later Oakhurst' as its lithic assemblage is characterized by relatively large scrapers with adze-like retouch (Mitchell 1993a:120-121). Faunal material recovered from layer GS proved to be unsuitable for isotopic analysis, however, other cultural and environmental information from this layer may prove

useful in determining a possible isotopic trend between the previous early, and subsequent middle, Holocene layers.

CSL-LR (7230±80 BP - Pta-5171), CSL-UP (6910±80 BP - Pta-5162), and CCL (6140±100 BP - Pta-5158) represent various stages of the interior Wilton Industry at Tloutle. Layer CSL-LR is an early Wilton phase with an increased abundance of backed microltihs and the continued presence of 'late Oakhurst' type artefacts (Mitchell 1990:104, 1993a:121). This is followed by a classic Wilton Industry in layers CSL-UP and CCL that is represented by reduced scraper size and an increased abundance of microlithics and segments (Mitchell 1990:104, 1993a:121). Charcoal analysis suggests that a mesic woodland was in place throughout this phase, with some xerophytic elements present in layer CLS-LR (Esterhuysen 1996:88, 90; Esterhuysen and Mitchell *in press*). Fauna from all three layers reflects an abundant and highly diversified assemblage, containing small to large grazing and browsing ungulates, ostrich bones and shells, and small mammals (Plug 1993).

Ha Makotoko (HM) And Ntloana Tsoana (NT)

Local Environment

The rock shelters of Ha Makotoko (29°20'S; 27°48'E) and

Ntloana Tsoana (27°19S; 27°49'E) lie within 2km of each other on the south bank of the Phuthiatsana-ea-Thaba Bosiu River, with the front of both shelters opening to the northwest (Mitchell 1993b:40, 42). These two sites are situated approximately 22km northwest of Tloutle. At the lower attitude of ±1640m asl they fall within the Lesotho Lowlands (see Fig. 5.7), as defined by Bawden and Carroll (1968) and Guillarmod (1971). Other than a mean annual rainfall of 736mm recorded from a station near to Ha Makotoko (reported in Esterhuysen and Mitchell *in press*), very little local climatic information is available for this area. A general overview of climatic information for Lesotho has been previously discussed (see Local Environment for Tloutle) and, therefore, will not be repeated here.

The natural vegetation of the low lying Phuthiatsana-ea-Thaba Bosiu River Basin is comprised of a mixed-sourveld *Cymbopogon-Themeda* grassland (Veld Type 48, Acocks 1988), with trees and shrubs restricted to gullies and valleys (Guillarmod 1971; Mitchell 1993b). Woody vegetation immediately surrounding the sites appears to reflect dry/cooler conditions than those seen in the Roma Valley. This may be related to a climatic inversion factor occurring in the Phuthiatsana-ea-Thaba Bosiu River Valley (Esterhuysen and Mitchell *in press*). The foothills near Ha Makotoko and Ntloana Tsoana bear a grassland that is transitional between *Cymbopogon-*

Themeda and a highland sourveld (Veld Type 56, Acocks 1988). The historical pattern for the presence of grazing and browsing ungulates in the lowlands is similar to that of Tloutle, as is the present trend towards an increase in xerophytic vegetation due to over-cultivation (Mitchell 1993b:40).

Background To Sites

Both sites were excavated in 1989 by Peter Mitchell (1993b, 1994). At Ha Makotoko excavations were carried out to bedrock over six square metres (Fig. 5.10). The natural stratigraphical units (Fig. 5.11) have been grouped into six layers (Mitchell 1993b). The top three layers, SS, DC, and TS, contain little artefactual evidence, except for a few adiagnostic pot sherds and remains from a 19th century occupation (Mitchell 1993b:40). An early Holocene occupational sequence is represented in the lower three layers (Mitchell 1993b, 1994).

Excavation at Ntloana Tsoana took place in three areas of the shelter (Fig. 5.12): front (L/M14), middle (K9/10), and rear (M4) (Mitchell 1993b). In total five square metres were excavated. Most of the material for this study came from K9/K10. This is the most complete sequence of the three, and has been divided into seven layers (Fig. 5.13a). Further material for this study

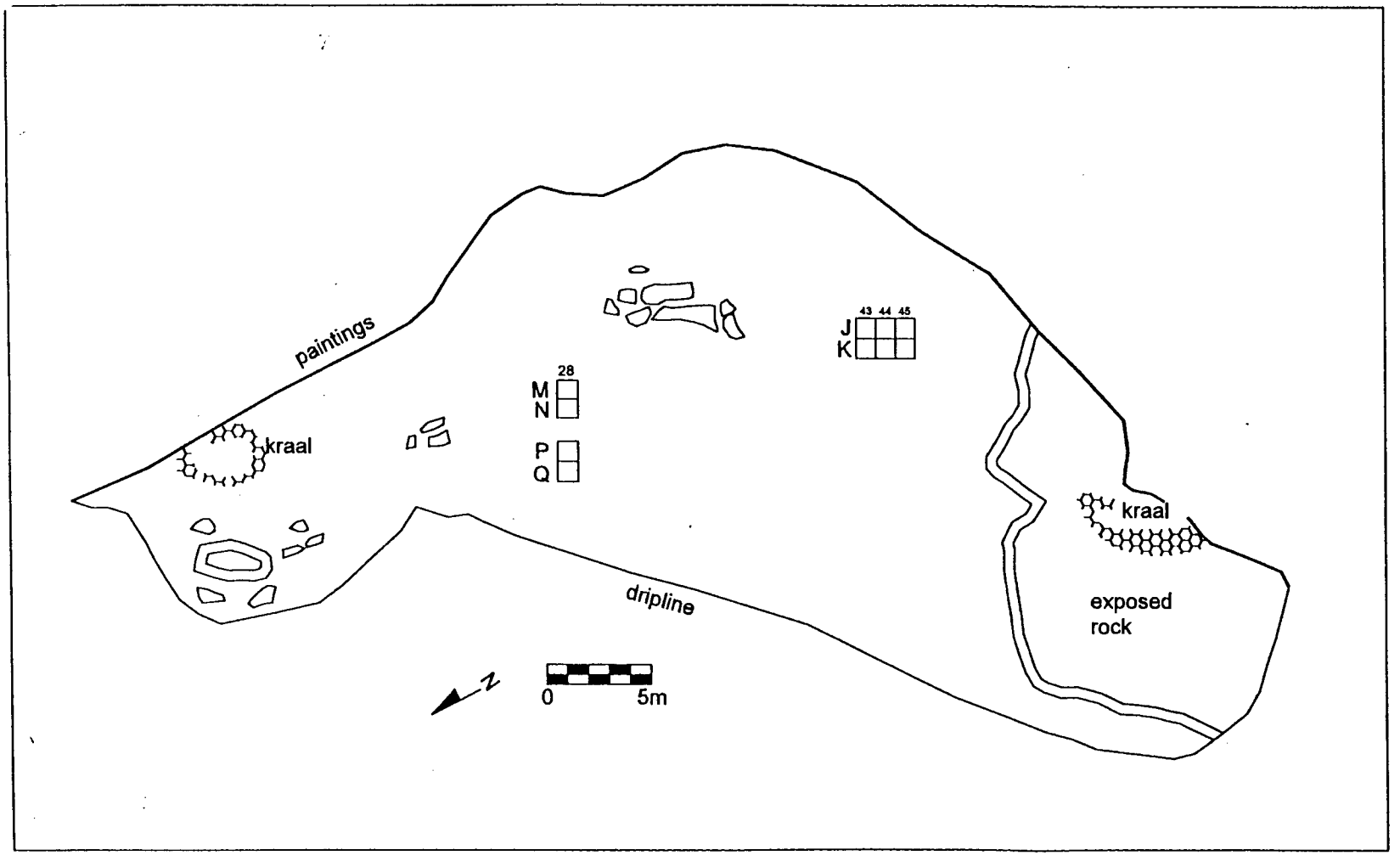

Figure 5.10 Ha Makotoko site plan and location of excavation (redrawn from Mitchell 1993b)

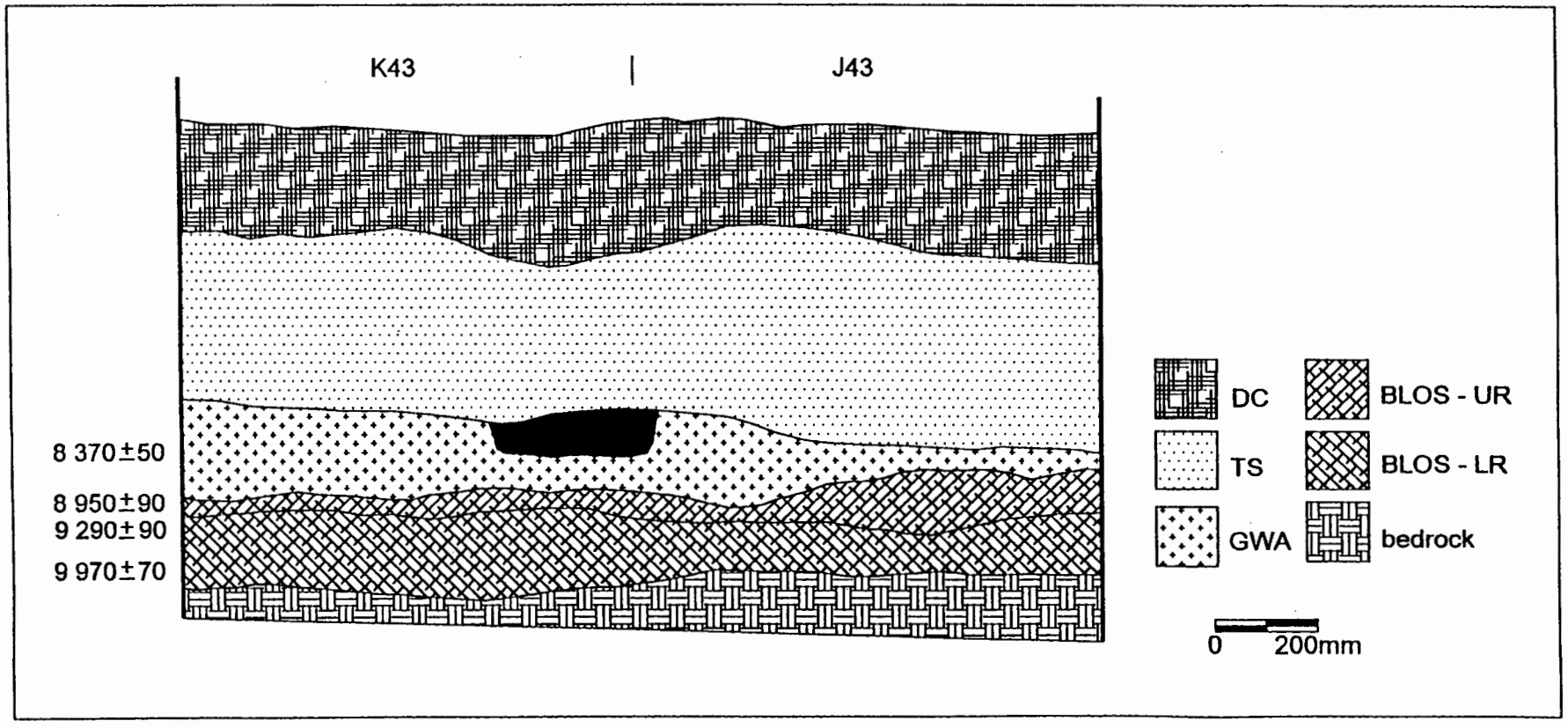

Figure 5.11 Ha Makotoko stratigraphic sequence (redrawn from Mitchell 1993b)

came from excavation L/M14 (Fig. 5.13b). Bedrock was not reached in any of the squares. An occupational pattern comparable to that of Ha Makotoko is present. The upper layer, SS, consists of small artefact scatters of recent Holocene age, none of which are chronologically diagnostic, while cultural material from the lower layers are associated with the early Holocene (Mitchell 1993a:43, 44). At both Ha Makotoko and Ntloana Tsoana the later sequences are capped by layer TS, a thick horizon of sterile aeolian sediments (Mitchell 1993b:43).

Context Of Faunal Samples

The Later Stone Age sequences for both Ha Makotoko and Ntloana Tsoana overlap; for this reason the sequences will be discussed together. An Oakhurst Industry, with a lithic assemblage comparable to those of Tloutle and Rose Cottage Cave, is associated with layer **BLOS-LR (9970±90 BP - Pta-5205 and 9290±90 BP - Pta-5204)** at Ha Makotoko, and at Ntloana Tsoana with **Area L/M14** and layer **BLOS (12 110±12 BP - Pta-5236, 10 200±100 BP - Pta-5208, 9420±110 BP - Pta-5237, and 9690±120 BP - Pta-5207)** (Mitchell 1993b, 1994). During this period opaline appears to have been the dominant raw material used at the Phuthiatsana-ea-Thaba Bosiu Basin sites, rather than hornfels as was the case at Rose Cottage Cave. Mitchell (1994:89) suggests that this may be

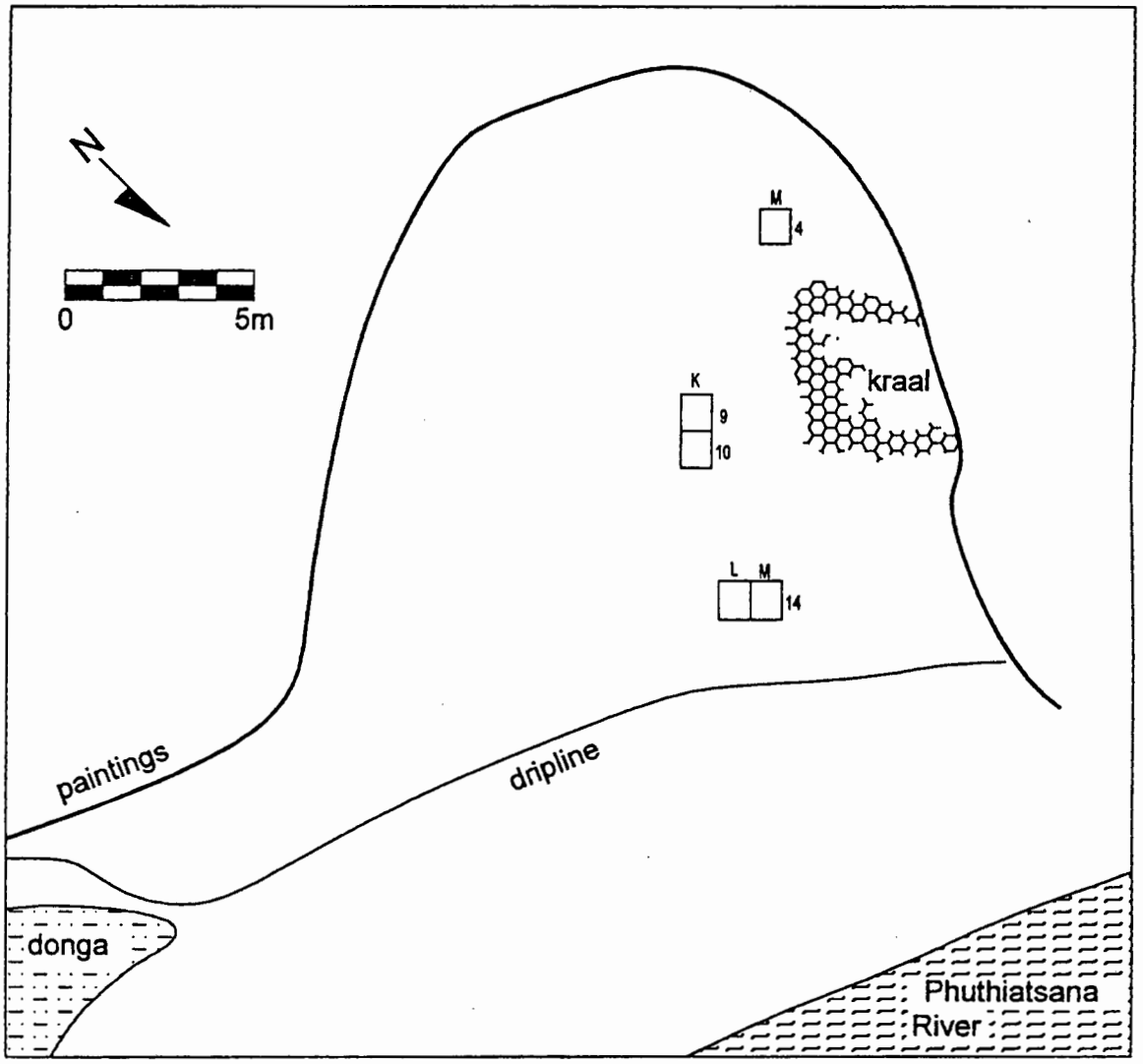

Figure 5.12 Ntloana Tsoana site plan and location of excavation (redrawn from Mitchell 1993b)

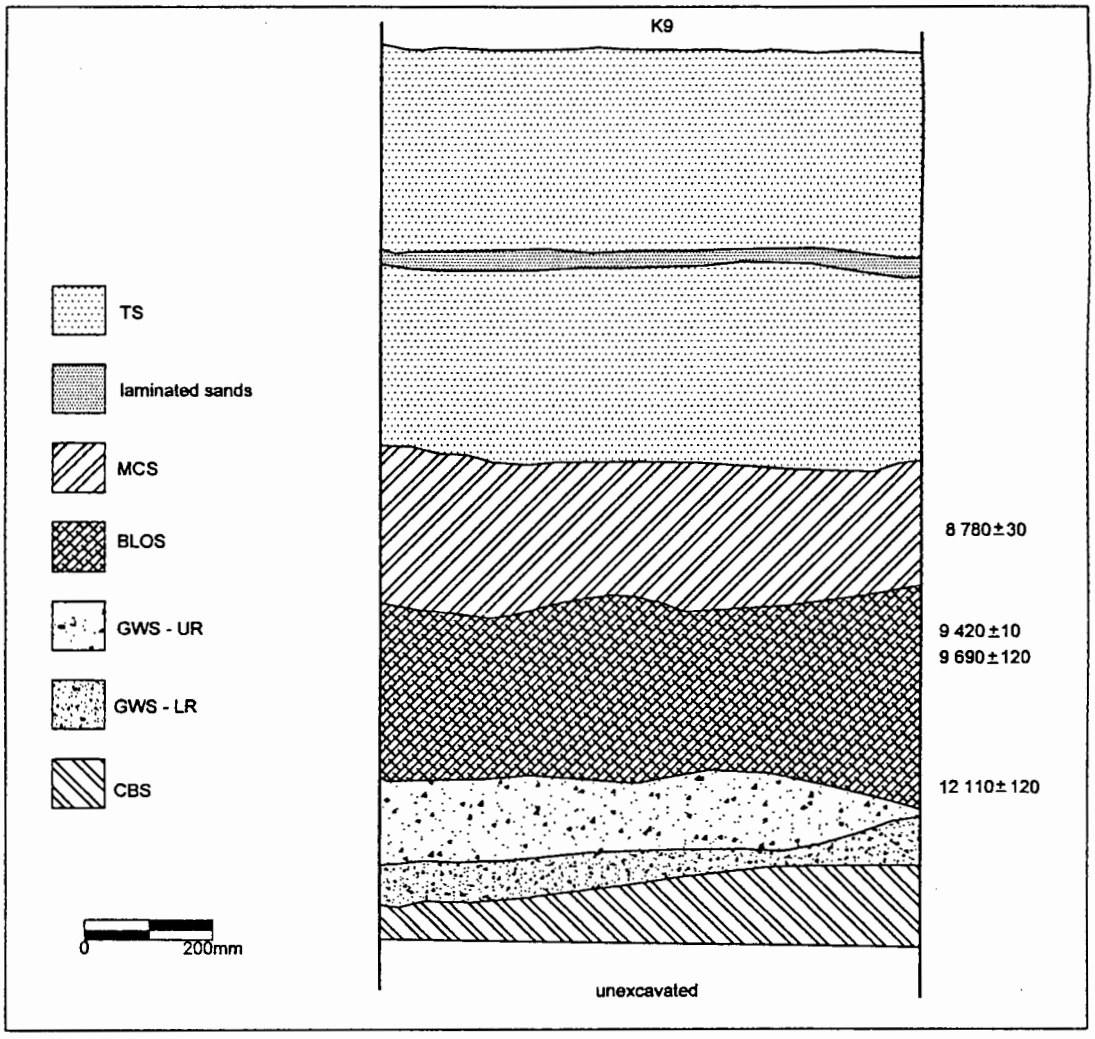

Figure 5.13a Ntloana Tsoana stratigraphic sequence for excavation K9 (redrawn from Mitchell 1993b)

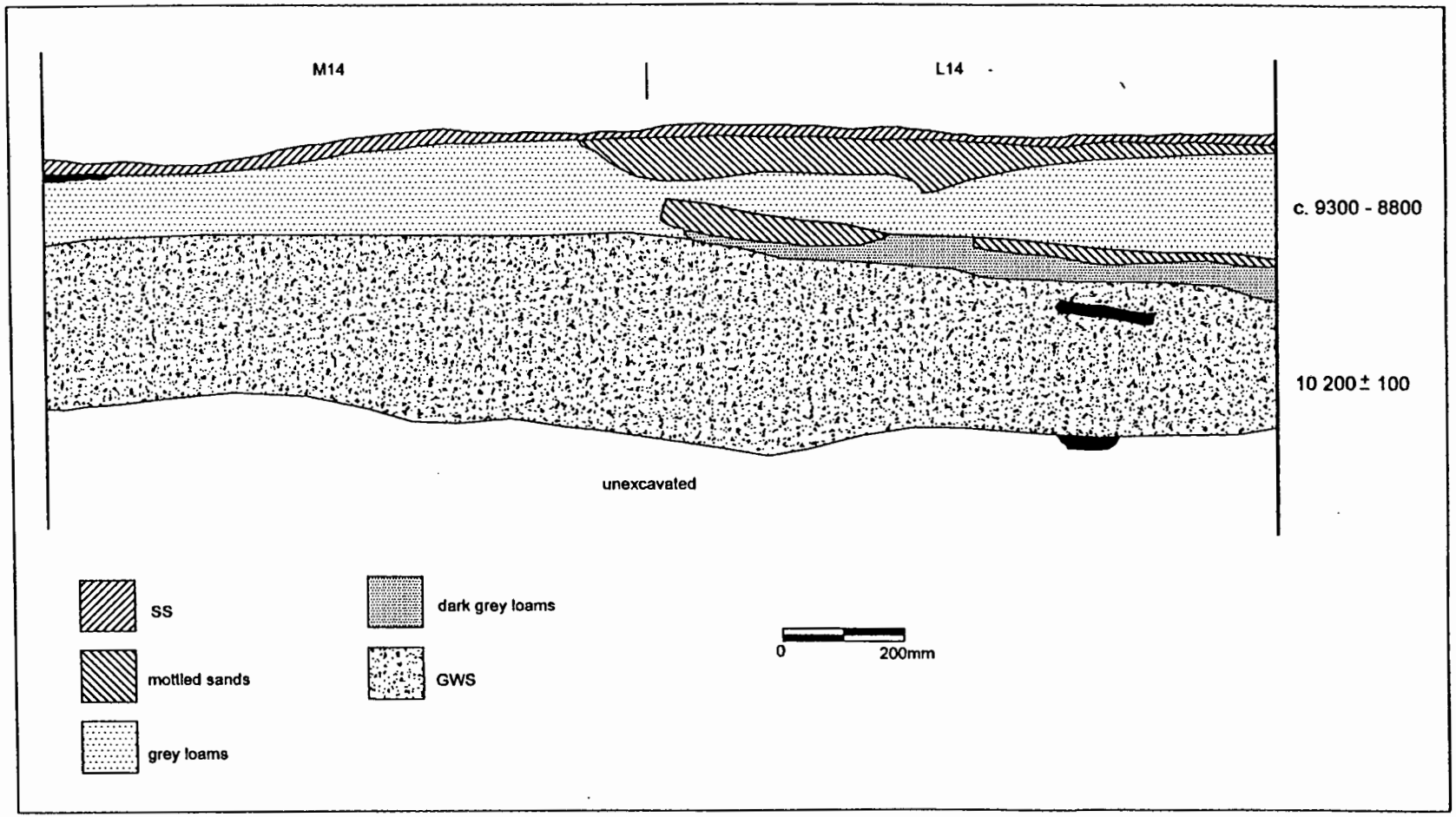

Figure 5.13b Ntloana Tsoana stratigraphic sequence for excavation L/M14 (redrawn from Mitchell 1993b)

"a marker to assert a social identity distinct from groups on the west of the Caledon River, where opalines are not found". Other artefacts of note come almost exclusively from Ha Makotoko, and include ostrich eggshell beads and ochre (Mitchell 1993b). The woody plant taxa identified from charcoal recovered from Ha Makotoko and Ntloana Tsoana were generally more suited to xerophytic conditions (Esterhuysen 1996:94). A wide variety of fauna was present, with large grazing and browsing ungulates dominating the assemblages at both sites. Both woodland and open grassland species of ungulates are present (Mitchell 1994:54).

The 'later Oakhurst' layers at Ha Makotoko, **BLOS-UR (8950±80 BP - Pta-5192) and GWA (8370±80 BP - Pta-5191)**, and at Ntloana Tsoana, **MCS (8780±30 BP - Pta-5238)** and **Area L/M14**, differ from the earlier industry by the appearance of a large number of 'Woodlot scrapers' that are characterized by adze-like retouch along the lateral edges (Mitchell 1994:90). In the faunal assemblage from this period there is a shift away from large bovids and equids towards smaller bovids (Mitchell 1994:90).

Conclusion

A review of the Late Pleistocene and Holocene occupational sequences for the sites under study has provided a context from which faunal material and soil

organic matter have been sampled for stable carbon isotopic analysis. The terminal Pleistocence/early to middle Holocene Later Stone Age sequences from the sites Rose Cottage Cave, Tloutle, Ha Makotoko, and Ntloana Tsoana overlap. Thus, consideration of the entire suite of sites provides the possibility for a more complete and conclusive climatic sequence than would be possible from only one set of observations. Furthermore, the presence of charcoal, fauna, and sedimentological analyses provide independent means of assessing reconstructions of past environments and climates based on isotopic analysis. From this overview it also becomes apparent that although all the sites fall within a grassland biome, local environmental conditions, such as those of Ha Makotoko and Ntloana Tsoana valley, may need to be taken into consideration when interpreting the isotopic results from these sites.

CHAPTER 6

RESULTS AND DISCUSSION

This chapter is divided into three parts: Modern $\delta^{13}C$ Values For The Caledon River Valley, Isotopic Results From The Study Of Fauna From The Terminal Pleistocene To The Middle Holocene and Isotopic Results From The Study Of Fauna And Soil Sediments For The Last 2300 Years. The first part sets out to establish a modern stable carbon isotopic baseline for the study area. Since the environmental and human factors forming the modern grassland are known, variables influencing interpretation are examined. This forms an important cache of information from which to better understand and interpret environmental change in the past. In part two, the data from the terminal Pleistocene (ca. 14 000 - 10 000 BP), early Holocene (ca. 10 000 - 7000 BP), and middle Holocene (ca. 7000 - 4000 BP) at Rose Cottage Cave, Tloutle, Ha Makotoko, and Ntloana Tsoana are discussed and compared. The degree of overlap between occupations during this period is highlighted in order to assess the comparability or uniqueness of each data set. Additionally, the results from this study are compared with independent lines of palaeoenvironmental evidence from these and other sites within the region of the eastern Free State and Lesotho, and adjacent areas. The third part provides an analysis and discussion of results obtained from isotopic studies of both fauna and

soil organic matter from sites dated to the last 2300 years.

PART ONE

Modern $\delta^{13}C$ Values For The Caledon River Valley

The Caledon River Valley may be identified as a pure C_4 grassland, based on the general parameters of climate and rainfall set out by Vogel *et al.* (1978). It was necessary, however, to determine the modern isotopic signature for the immediate C_3 and C_4 vegetation for two reasons. First, to establish a set of modern values to serve as a datum for comparison, and secondly to isolate any local variations in $\delta^{13}C$ values.

Three species of grasses, *Themeda triandra, Eragrostis sp., Aristida congesta sp. congesta*, and an indeterminate species were collected from the north facing scrub and open grasslands just below Rose Cottage Cave. The analysis of all four species (n=12) provided a mean $\delta^{13}C$ value of -12.7±0.8‰ (Table 6.1). No apparent difference exists between grass values from scrub or open grassland sites. Three samples of woody vegetation collected from the same slope have a mean $\delta^{13}C$ value of -25.7±0.6‰ (Table 6.2). Overall, the mean C_3 and C_4 values for the Rose Cottage Cave area are similar to those recorded for South Africa as whole (Vogel *et al.* 1978).

Table 6.1 $\delta^{13}C$ values for modern grasses from the Ladybrand area

UCT #	Site	Species	Grass Part	$\delta^{13}C$ ‰
6019	Scrub Grassland	*Themeda triandra*	Inflorescence	-12.0
6020	Scrub Grassland	*Themeda triandra*	Stem	-12.1
6021	Scrub Grassland	*Themeda triandra*	Blade	-13.1
6022	Scrub Grassland	*Eragrostis sp.*	Inflorescence	-12.4
6023	Scrub Grassland	*Eragrostis sp.*	Stem	-11.7
6024	Scrub Grassland	*Eragrostis sp.*	Blade	-14.2
6025	Open Grassland	Indeterminate	Inflorescence	-12.0
6026	Open Grassland	Indeterminate	Stem	-12.6
6027	Open Grassland	Indeterminate	Blade	-12.2
6028	Open Grassland	*Aristida congesta sp. congesta*	Inflorescence	-12.4
6029	Open Grassland	*Aristida congesta sp. congesta*	Stem	-13.3
6030	Open Grassland	*Aristida congesta sp. congesta*	Blade	-14.0
			Mean $\delta^{13}C$	-12.6
			Std. Dev.	±0.8

The separate parts of the grass - inflorescence, stem,and blade - were independently analysed, as "bulk" and "concentrated" grazers have a preference for specific parts of the grass at different times of the year (Bell 1969; Vesey-Fitzgerald 1960). Grass blades may be slightly depleted in ^{13}C (mean=-13.3±0.8‰, n=4) compared to bulk feeders, or those preferring the inflorescence (mean=-12.2±0.4‰, n=4) or stem (mean= -12.4±0.7‰, n=4) (Table 6.3). If the pattern holds, then grazers favouring grass blades may have slightly more negative $\delta^{13}C$ values than bulk feeders or those preferring the inflorescence or stem. A larger sample size is needed to verify these results. It should be noted that the differences between these values are not great enough to suggest an isotopically different diet source, and therefore, will not mislead interpretation based on isotopic evidence.

Stable carbon isotopic data are unavailable for the present vegetation surrounding the western Lesotho sites. However, isotopic data does exist for the area surrounding Sehonghong in the eastern Lesotho Highlands. Grasses sampled by P. Mitchell (Oxford) and G. Hall (UCT) were analysed in the Archaeometry Unit, UCT. Ten specimens taken from the north facing slope at altitudes of 1725m (Plot A) and 1975m (Plot G) asl have a combined mean $\delta^{13}C$ value of -14.5±0.8‰ (Table 6.4). The 2‰ depletion in ^{13}C compared to that of the Ladybrand

Table 6.4 $\delta^{13}C$ values for modern grasses from eastern Lesotho at altitudes of 1725m (Plot A) and 1975m (Plot G) asl

UCT No.	Site	Species	Plant Part	$\delta^{13}C$ ‰
5914	Plot A6	*Aristida sp.*	Stem	-12.9
5918	Plot A10	*Aristida adscenionis*	Stem	-14.4
5922	Plot A14	*Tricholaena monachne*	Stem	-14.7
5928	Plot A20	*Chloris virgata*	Stem	-14.6
5933	Plot A25	*Eragrostis curvula*	Stem	-15.0
5996	Plot G5-8	*Aristida adscenionis*	Stem	-15.1
5998	Plot 9-12	*Cymbopogon sp.*	Stem	-14.2
6001A	Plot G13-16A	*Panicum sp.*	Stem	-15.9
6001B	Plot G13-16B	*Aristida sp.*	Stem	-14.1
6002	Plot 17-20	*Panicum sp.*	Stem	-14.0
			Mean $\delta^{13}C$	-14.5
			Std. Dev.	±0.8

area may arise from a combination of local factors, such as decreased evaporation and lower temperatures - resulting in higher CO_2 concentrations - and low fertile basaltic sediments associated with the eastern Lesotho Highlands. Given these micro-environmental influences, the present C_4 values for the Lowlands and Foothills of western Lesotho are considered to be closer to those for Ladybrand, as these areas are more climatically similar. However, environmental factors influencing the isotopic values of grasses from the eastern Lesotho Highlands may serve as an example from which to assess similarly depleted $\delta^{13}C$ values of fossil grazers from sites in the Lowlands and Foothills.

Due to the near extinction of ungulate herbivores in the Caledon River Valley, $\delta^{13}C$ values had to be extrapolated from those for the present vegetation. Assuming a diet-apatite difference of 12.5‰, grazers with a diet of 100% C_4 grasses would expected to have a value of -0.2±0.8‰ for apatite. If the diet-collagen difference is about 5‰, one would expect a value of -7.7±0.8‰ for collagen. Browsers with a 100% C_3 diet would demonstrate apatite and collagen values of -13.2±0.6‰ and -20.7±0.6‰ respectively. These predicted values are comparable to actual values recorded for modern herbivores from southern Africa (Vogel 1978; Lee-Thorp 1989). The ratio of C_3/C_4 vegetation contributing to the herbivore diet, as expressed in this study, is based on 1.3‰ enrichment

for every 10% of C_4 biomass consumed; assuming a 13‰ difference between 100% C_3 and C_4 diets. This characterization of the isotopic values for modern grazers is derived from a fairly limited sample of modern grasses, and so provides only a rough guide.

The fossil fuel effect has resulted in modern values being depleted in ^{13}C by 1.5 to 2‰, in comparison to pre-industrial plants and animals. For this reason the 100% C_3 and C_4 baseline presented here incorporates a 2‰ enrichment factor. In the second part of this chapter, "present" environmental conditions of the study area will be represented by this proposed baseline. Taking into account the standard deviation, fossil apatite values for a pure C_4 grazer should fall within a range of 1.2 to 2.6‰, and for pure C_3 consumers between -11.8 and -10.6‰. Values greater than 2.6‰ have been found in this study, and as these values are more enriched than any other grazer apatite results published for the last 14 000 years for South Africa (for example Thackeray and Lee-Thorp 1992; Lee-Thorp and Beaumont 1995), they were initially thought to be the result of diagenetic alteration. The enriched values, however, were subsequently seen to be acceptable because: 1) there is a ca. 8‰ difference between collagen and apatite from the same individual or between grazing species with similar habitat preference, and 2) there exists a ca. 13‰ difference between apatite values for pure

grazers and browsers. Collagen was generally not well preserved, but in samples where it was recovered the collagen had acceptable C/N ratios.

Summary

A modern baseline of -12.7±0.8‰ for C_4 and -25.7±0.6‰ for C_3 was set up from modern vegetation sampled from sites in the eastern Free State. Although no modern samples were processed from the area surrounding the western Lesotho sites, grasses analysed from similar altitudes in the Lesotho Highlands proved to be more depleted (2‰) than the Free State samples. The difference in values was accredited to higher concentrations in CO_2; a microclimatic situation that may serve as an analog for similar depletions in the past. Further variation in values was picked up within the grass structure itself. These differences, however, were not great enough to be misconstrued as a C_3/C_4 shift in vegetation.

Modern faunal values were extrapolated from the values for the present vegetation. Grazers with a diet of 100% C_4 grasses would have a values of -0.2±0.8‰ for apatite, and -7.7±0.8‰ for collagen. Browsers with a 100% C_3 diet would demonstrate values of -13.2±0.6‰ for apatite, and -20.7±0.6‰ for collagen. Due to the fossil fuel effect, however, all values would need to be enriched by 2‰.

PART TWO

Isotopic Results From The Study Of Fauna From The Terminal Pleistocene To The Middle Holocene

The mean $\delta^{13}C$ values and the dietary inferences for grazers from the terminal Pleistocene through to the middle Holocene levels at Rose Cottage Cave, Tloutle, Ha Makotoko, and Ntloana Tsoana are presented in Table 6.5. These mean values have been plotted out in Fig. 6.1, which illustrates that fluctuations in isotopic values have occurred during this period. The raw data from each of the sites are found in Appendix I sections A, C, D, and E.

In general, results from the terminal Pleistocene levels at Rose Cottage Cave indicate that it was a period in which the percentage of C_4 grasses incorporated into the diet of grazers increased to that of the present, while that of Tloutle, Ntloana Tsoana, and Ha Makotoko remained slightly below this level. At ca. 13 400 BP mean values of -2.5±1.3‰ (n=7) for Rose Cottage Cave and -4.5‰ (n=2) for Tloutle suggests that the consumption of C_4 grasses by grazers was below that of present. Data from Appendix I section A and C, indicate substantially depleted values of -6.1‰ for *E. quagga* (UCT 5494) and -4.6‰ for *A. buselaphus* (UCT 5132) from Rose Cottage Cave, and -7.3‰ for *D. dorcas* (UCT 5459) from Tloutle. These values reflect diets that contained less than 50%

Table 6.5 Mean $\delta^{13}C$ apatite values for grazers from terminal Pleistocene to middle Holocene levels

Site	yr BP ± SD	Level	n	Min. (‰)	Max. (‰)	Mean $\delta^{13}C$ ± SD (‰)	Approximate % C_4 In Diet
RCC	5970 ± 70	Pt-UP	8	0.8	3.8	2.4 ± 1.0	100
TL	6140 ± 100	CCL/CSL-UP	3	0.7	1.8	1.4 ± 0.7	100
TL	6910 ± 80	CSL-UP	3	3.1	3.8	3.5 ± 0.5	100
TL	7230 ± 80	CSL-UP/LR	3	-7.3	-4.6	-6.4 ± 1.3	30
RCC	7630 ± 80	Pt-LR	2	-2.8	-2.3	-2.6	60
RCC	8160 ± 70	Ja	7	1.7	2.6	2.2 ± 0.5	100
RCC	8350 ± 70	Ph	5	-2.7	0.6	-0.8 ± 1.1	80
HM	8370 ± 80	GWA	2	-1.9	-0.7	-1.3	70
RCC	8380 ± 70	JaG	2	-0.9	-0.6	-0.8	80
RCC	ca.8500	Cm	5	-3.7	-1.1	-1.8 ± 0.9	70
RCC	8614 ± 38	Ha	4	-10.6	-2.0	-5.4 ± 1.6	40
NT	8780 ± 30	MCS	1			-2.7	60
TL	ca.9000	BS	1			-4.6	50
NT	ca.9300-8800	Area L/M14	2	-7.3	-6.2	-6.6	30
HM	9290 ± 90	BLOS-UR/LR	2	-10.9	-3.8	-7.4	20
HM	9970 ± 90	BLOS-LR	3	-3.0	-0.9	-2.3± 1.0	60
NT	10 200 ± 100	Area L/M14	2	-3.0	-0.1	-1.6	70
RCC	ca.12 000	DCM	1			2.0	100
NT	12 110 ± 120	BLOS	1			-10.4	0
RCC	12 690 ± 120	DB-UP	3	0.4	2.3	1.6 ± 0.9	100
RCC	13 360 ± 150	DB-LR	7	-6.1	-1.6	-2.5 ± 1.3	70
TL	ca.13 400	BC	2	-7.3	-1.7	-4.5	50

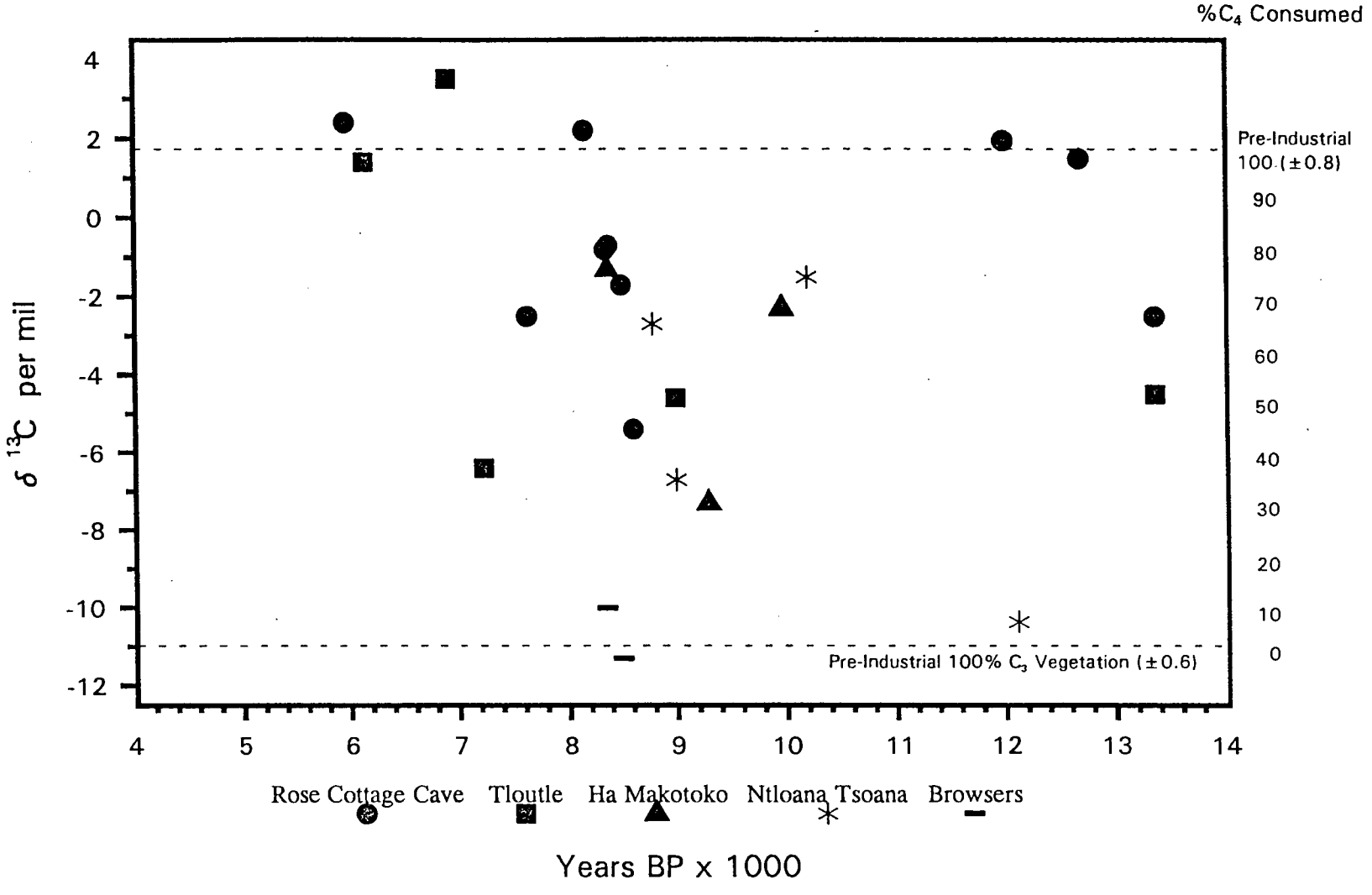

Figure 6.1: Plotted data from Table 6.5: Mean $\delta^{13}C$ values for herbivores for 13 500 - 5800 BP

C_4 grasses. Overall, grazers from Rose Cottage Cave and Tloutle consumed 30% and 50% less C_4 grasses respectively, relative to modern grazers with 100% C_4 diets. In the next two levels from Rose Cottage Cave, the mean $\delta^{13}C$ values for 12 690±120 BP (1.6±0.9‰, n=3) and ca. 12 000 BP (2.0‰, n=1) indicate that C_4 grasses now made up 90 to 100% of the diet. In contrast, although increasing in abundance, grazers from Ntloana Tsoana at 10 200±100 BP (mean=-1.6‰, n=2), and from Ha Makotoko at 9970±90 BP (mean=-2.3±1.0‰, n=3) had diets containing only 60 to 70% C_4 grasses.

The anomalous value of -10.4‰, reflecting negligible C_4 intake, for an *E. quagga* (UCT 5479) at 12 110±120 BP from Ntloana Tsoana has been included, as it may indicate the continued presence of C_3 grasses within the feeding range of *Equus*. This specimen was not considered to be a misidentification of a species of browser, as in this study other specimens of *Equus* are shown to have substantially depleted $\delta^{13}C$ values.

Following the increase in C_4 grasses towards the end of the terminal Pleistocene, C_3 grasses made a significant contribution to the diet of grazers in the early to middle Holocene levels at all four sites (see Fig. 6.1). Mean $\delta^{13}C$ values of -7.4‰ (n=2) for Ha Makotoko at 9290±90 BP, -6.6‰ (n=2) and -2.7‰ (n=1) for Ntloana Tsoana ca. 9300 - 8800 BP and at 8780± 30 respectively,

and -4.6‰ (n=1) for Tloutle ca. 9000 BP, provide evidence that C_4 grasses constituted between 20 to 50% of the diet. Rose Cottage Cave, supports this pattern as C_4 grasses contributed only 40% (mean=-5.4±1.6‰, n=4) to the diet of grazers at 8614±38 BP. The values of -10.9‰ for a *C. gnou* at 9290±90 BP (UCT 5464) from Ha Makotoko, and -10.6‰ for a *D. dorcas* at 8614±38 BP from Rose Cottage Cave are considered reliable indicators of environmental conditions. These two species of grazers have been known to browse during adverse conditions when suitable grasses are not available (Lynch 1983:173; Smithers 1992:155, 163). If prolonged, this addition of C_3 vegetation to the diet would lower the isotope values. However, since species that are exclusively grazers also display substantially depleted values (see Appendix I), although not to the same extent, it is proposed that C_3 grasses, rather than woody browse, contributed more substantially to their diet.

The mean $\delta^{13}C$ values for levels encompassing the next four hundred years at Rose Cottage Cave, ca. 8500 BP (-1.8±0.9‰, n=5), 8380±70 (-0.8‰, n=2), and 8350±70 (-0.8±1.1‰, n=5), as well as for Ha Makotoko at 8370±80 (-1.3‰, n=2) show that the percentage of C_4 grasses present in the diet of grazers increased to between 70 and 80%. By 8160±70 BP values for Rose Cottage Cave had reached 100% (mean=2.2±0.5‰, n=7). A fluctuation in the C_4 component occurred once again in levels dating to

7630±80 BP at Rose Cottage Cave (mean=-2.6‰, n=2) and 7230±80 BP at Tloutle (mean=-6.4±1.3‰, n=3). At these times C_4 grasses decreased to 60% and 30% respectively. This was followed by a shift to 100% C_4 grasses, as indicated by the mean $\delta^{13}C$ values for Tloulte of 3.5±5‰ (n=3) at 6910±80 BP and 1.4±0.7‰ (n=3). at 6140±100 BP, as well as that of 2.4±1.0‰ (n=7) for Rose Cottage Cave at 5970±70 BP.

Interpretation Of $\delta^{13}C$ Values

Results from this study indicate a general trend from ca. 13 500 - 5800 BP towards temperatures and seasonality of rainfall analogous to the present in the Ladybrand area and western Lesotho by ca. 6000 BP (see Fig. 6.1). However, this was not a linear development. During this period the principally C_4 diet of the grazers is consistent with a predominantly summer rainfall regime having temperatures above the C_4 threshold. But fluctuating $\delta^{13}C$ values suggest that at various intervals conditions were sufficiently cold during the growth season to foster the development of a C_3 component.

Depleted $\delta^{13}C$ values, of between 3 and 8‰, for grazers at both Rose Cottage and Tloutle indicate that at ca. 13 400 BP temperatures would have been cool enough to support C_3 grasses some parts of the migratory route. As a similar range of $\delta^{13}C$ values are recorded for both

localities, this could possibly reflect a extension of the rainfall season into the cooler late summer/autumn months. Although it is generally accepted that cooler temperatures accompanied the terminal Pleistocene, the $\delta^{13}C$ values at ca. 13 400 BP have a 50 to 70% C_4 content. This most likely reflects a warming trend near the terminus of the LGM, rather than a continuation of cooler glacial conditions.

Results for Rose Cottage Cave from 12 690±120 BP and ca. 12 000 BP are comparable with those for modern grazers. These values are consistent with a summer rainfall pattern and growth temperatures above the C_4 threshold. In relation to Tloutle, a general increase in C_4 grasses is also recorded for Ntloana Tsoana and Ha Makotoko at ca. 10 000 BP. However, the 30 to 40% C_3 grasses in the diet of grazers suggest that slightly cooler temperatures prevailed in Lesotho, such that grasses depleted in ^{13}C persisted at altitudes low enough to be utilized by grazers. This situation may have been similar to that presently found in the eastern Lesotho Highlands where conditions are wetter and cooler, and grasses are slightly depleted in relation to the eastern Free State and western Lesotho.

The first part of the early Holocene, ca. 9300 - 8600 BP, is marked by an increased percentage of C_3 grasses at all three western Lesotho sites. Since

similar values occur for Rose Cottage Cave at ca. 8600 BP, the rise in C_3 grasses is best attributed to an extended rainfall season. Following this episode, data from Rose Cottage dating between ca. 8400 and 8100 BP indicate a return to summer rainfall conditions and temperatures analogous to the present. A corresponding increase in C_4 grasses is recorded for Ha Makotoko at 8370±80 BP, but it not clear whether a 100% C_4 grassland was achieved by ca. 8100 BP. There was another excursion towards lower growth season temperatures at Rose Cottage Cave ca. 7700 BP and at Tloutle 7300 BP. The approximately 400 year gap separating Tloutle and Rose Cottage Cave precludes interpreting the decrease in C_4 grasses as either an extended summer rainfall season or as lower temperatures during that season. In either case, temperatures in Lesotho were mostly likely cooler than those to the west, as $\delta^{13}C$ values are at least 2‰ more depleted, with C_4 grasses comprising only 30% of grazers' diet compared to 60% for those from Rose Cottage Cave.

The $\delta^{13}C$ values for the middle Holocene levels from Tloutle, at 6910±80 BP and 6140±100 BP, and Rose Cottage Cave, at 5970±70 BP, are consistent with a 100% C_4 grassland. For the western Lesotho sites, this is the first time that a pure C_4 grassland is indicated. Although the rainfall season for Rose Cottage Cave and western Lesotho probably followed a similar trajectory

throughout the sequence, it seems that it was not until the middle Holocene that temperatures for Lesotho became comparable to those of the present growth season. Conditions favouring a 100% C_4 grassland appear to persist throughout the later Holocene.

Related Palaeoenvironmental Evidence

Interpretation of environmental and climatic shifts for the late Pleistocene through to the middle Holocene, derived from this stable carbon isotopic study, can be compared with results from independent palaeo-environmental studies. Two sets of independent data exist. The first comprises charcoal, fauna, and sediments analysed from Rose Cottage Cave, Tloutle, Ha Makotoko, and Ntloana Tsoana. The second is derived from a more regional data set. It is necessary to draw on these multiple lines of evidence from "different dimensions of the archaeological record" and from "different ranges of background knowledge" in order to determine the significance of the evidence, as it is highly unlikely that independent sets of evidence could all "incorporate compensatory errors" (Wylie 1993:25).

Charcoal, Fauna, And Sediments From The Study Sites

Factor analysis carried out on the Rose Cottage and Tloutle charcoals provides a relative index of

temperature and moisture change for the terminal Pleistocene to middle Holocene (Esterhuysen 1996). This index offers an effective means of comparing the charcoal and isotopic data as it allows for level-by-level comparison (Smith and Esterhuysen in prep.). Overall the isotopic and charcoal data support one another (Fig. 6.2). The presence of both C_3 grasses and 'macchia-type' woody vegetation ca. 13 400 BP - 12 690 BP strengthens the assertion of cooler than present growth temperatures at this time. A single discrepancy exists between the isotopic and charcoal data from Rose Cottage Cave dated to 12 690±120 BP. This may be a consequence of the fact that when the charcoals were analysed no distinction was drawn between upper DB (12 690±120 BP) and lower DB (13 360±150 BP) (Esterhuysen pers. comm.), and any possible warming event in upper DB would have been masked. Lower than present growth temperatures at Ntloana Tsoana ca. 10 200 BP, is upheld by a similar interpretation for the charcoal. The general isotopic trend between ca. 13 500 - 10 000 BP towards increased C_4 grasses, and therefore warmer and more summer rainfall conditions, is not evident in the available charcoal data. For the early to middle Holocene both the isotopic and charcoal data provide evidence of the following growth temperatures:

- cooler than present ca. 10 200 - 8600 BP

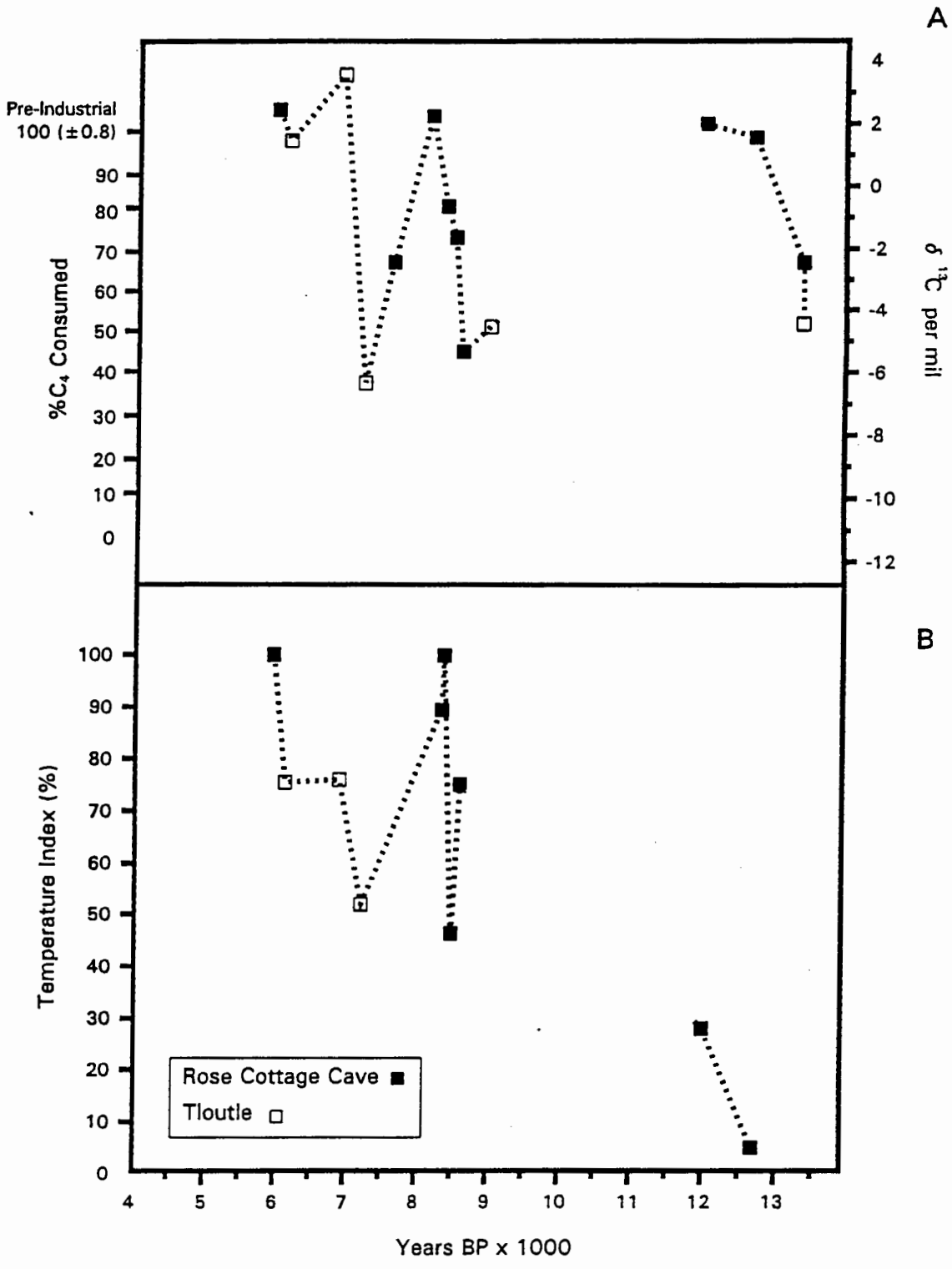

Figure 6.2 Plotted data from Table 6.1 and temperature index from charcoal factor analysis (Esterhuysen 1996)

- becoming warmer ca. 8400 - 8100; analogous to the present by ca. 8100 BP
- cooler than present ca. 7200 BP
- analogous to the present ca. 6900 and ca. 5900 BP.

After ca. 10 000 BP charcoals studied from the early Holocene layers at Ha Makotoko and Ntloana Tsoana did not provide evidence of change in woody vegetation. This was thought to be the result of "microclimatic conditions operating on and near the banks of the PTB (Phuthiatsana-ea-Thaba Bosiu) river" (Esterhuysen 1996:85). These conditions are seen to be responsible for producing woody vegetation that is insensitive to climatic fluctuation (Esterhuysen 1996:86). The high resolution isotopic data from these sites, with support from Rose Cottage Cave and Tloutle, indicate that, unlike the woody vegetation, grasses in this situation are sensitive to climatic changes and may potentially be a more effective indicator of climatic fluctuation.

Interpretations of sedimentological (Butzer 1984(a)(b)) and faunal data (Engela and Plug 1992; Plug 1993) concur with that of the isotopes and charcoal. Sedimentological analysis from Rose Cottage Cave indicates that between ca. 11 000 - 7300 BP the climate was initially cool, then warming towards that of present, with conditions wetter than the terminal Pleistocene and the present occurring at 8600±100 BP (Pta-2067) and again at 6850±45 BP (GrN-5299) (Butzer

1984a:57). The macro-fauna assemblages from the terminal Pleistocene levels at Rose Cottage Cave and Tloutle show low species diversity and numbers (Plug and Engela 1992; Plug 1993), "probably due to low primary production during these cooler periods" (Plug and Engela 1992:18). The early Holocene contained a higher component of woody vegetation, as inferred from the presence of browsers, and animals preferring wooded environments, such as vervet monkey and leopard (Plug and Engela 1992; Mitchell 1993b; Plug 1993; Engela 1995). As grazers still form a large portion of the faunal assemblage and the isotopic results reflect a predominately C_4 diet, it is most likely that the vegetation during this period was probably a wooded grassland. In the subsequent period, ca. 7600 - 5800 BP, the faunal assemblage from Rose Cottage is dominated by species associated with open grasslands; conditions that probably formed under a climate similar to that of the present (Wadley pers. comm.).

Regional Comparison Of Palaeoenvironmental Evidence

At present there are only a small number of terminal Pleistocene to middle Holocene sequences from other areas of the eastern Free State and Lesotho with

comparable palaeoenvironmental data. For the terminal Pleistocene, pollen studies from the eastern Free State sites of Craigrossie and Cornelia, in the north, and Aliwal North, to the south, show the development of cool mesic conditions prior to ca. 10 000 BP. This is interpreted as there being both summer and winter rainfall present in the region (Coetzee 1967; Scott 1989, 1990). In general this cooler pattern supports the terminal Pleistocene results from this study. For the higher altitude site of Cornelia, Scott's (1986) suggestion of cooler summer rainfall temperatures ca. 12 600 BP, may correspond to the low growth temperatures and altitudinal expansion of C_3 grasses proposed for western Lesotho ca. 12 000 BP. A warm and dry phase is noted at Aliwal North between ca. 11 650 - 11 250 BP (Coetzee 1967), and during approximately the same period at Craigrossie (Scott 1986). This may relate to the warmer growth temperatures for Rose Cottage Cave ca. 12 000 BP. The climatic variation seen between lower and higher altitude sites at this time, may provide evidence that higher altitudes are more sensitive to smaller decreases in temperatures and that these areas did not recovered as quickly from the LGM.

Extending the survey slightly further afield, charcoal studies from Ravenscraig, northern Eastern Cape, indicate that, relative to the Holocene, conditions ca. 10 000 BP were cooler and moister (Tusenius 1986,

1989). This is in keeping with the isotopic data of cooler than present growth temperatures for Ntloana Tsoana and Ha Makotoko at approximately the same time.

Evidence available for the early to middle Holocene comes from two localities in the eastern Lesotho Highlands. Peat and sedimentological studies from the Upper Mashai River catchment area indicate conditions wetter than present at 8650±80 BP (Pta-359) and 7280±150 BP (Q-1156); temperature was undetermined (Carter 1976: 198). These periods of increased moisture are comparable to that recorded by Butzer (1984a:57) for Rose Cottage Cave at ca. 8600 and 6800 BP. Marker's (1994) sedimentological studies in the Sani Top area suggest that conditions ca. 13 500 - 9000 BP were relatively warm and moist, followed by a period of low precipitation and cool temperatures ca. 9000 - 5000 BP. Low resolution dating prevents any finer scale comparison with sequences in this study.

Comparative isotopic data from the interior of southern Africa comes from the sites of Equus Cave and Wonderwerk Cave, Northern Cape. The $\delta^{13}C$ values of ungulate herbivores from both sites indicate that the terminal Pleistocene and Holocene grasslands were predominately C_4 in nature (Thackeray and Lee-Thorp 1992; Lee-Thorp and Beaumont 1995). However, at Equus Cave between ca. 12 000 - 8000 BP the values for grazers are

relatively depleted compared to modern values. As discussed in Chapter four, these values have been interpreted as reflecting lower growth season temperatures and possibly year round rainfall (Lee-Thorp and Beaumont 1995). This is in keeping with the findings for the Caledon River Valley for the terminal Pleistocene to early Holocene, and suggests that the extended season of rainfall may have been a larger regional phenomenon.

Summary

The $\delta^{13}C$ values of ungulate grazers recovered from the sites Rose Cottage Cave, Tloutle, Ha Makotoko, and Ntloana Tsoana have provided reliable and comparable results, for the purpose of palaeoenvironmental reconstruction. Within the context of the well dated cultural sequences spanning the terminal Pleistocene to middle Holocene, a relatively high resolution palaeoenvironmental sequence for the Caledon River Valley area was obtained. On a gross scale, over the period ca. 13 500 BP - 5800 BP, climatic conditions became increasingly more like those of the present, yet on a more refined scale significant fluctuations were discernible. Interpretation of these fluctuations provided the following index of change:

ca. 13 400 BP - cool conditions with a possible extension of summer rainfall,

ca. 12 690 - 10 000 BP - rainfall and temperatures comparable with those of today, although temperatures in Lesotho were slightly cooler than the eastern Free State,

ca. 9300 - 8600 BP - cooler, with possible extension of summer rainfall,

ca. 8400 - 8100 BP - conditions analogous to those of the present,

ca. 7600 - 7200 BP - cooler

ca. 6900 - 5900 BP - conditions analogous to the present.

These results were found to be consistent with independent sources of data. It was noted, however, that in certain environmental conditions isotope analysis may be more sensitive when identifying environmental changes. This is potentially evident for the early Holocene levels at Ha Makotoko and Ntloana Tsoana where no change was registered in the charcoals studies, but other sources of palaeoenvironmental data are needed to substantiate this possibility.

PART THREE

Isotopic Results From The Study Of Fauna And Soil Organic matter For The Late Holocene

Palaeoenvironmental data for this part of the study is confined to the last 2300 years and is based predominantly on materials recovered from Rose Cottage Cave and Twyfelpoort. Results of the carbon isotopic analysis of grazers from Rose Cottage Cave, combined

with those for the soil organic matter within the vicinity of the site, provide information for layers dating to around 2200 BP, 1600 BP, 700 BP, and 500 BP. Further isotopic results from the site of Twyfelpoort contribute data for the period of ca. 1900 BP.
In order to interpret the isotopic data certain factors need to be considered. First, as proposed in Chapter four, $\delta^{13}C$ values for soil organic matter should reflect the micro-environment, whereas those for the migratory grazers should incorporate a more regional picture. Secondly, the presence of domesticated grazers, associated with archaeological evidence for farming from sites in the Ladybrand area ca. 500 BP (Wadley 1996), may have implications for the alteration of a primarily C^4 grassland to one of more C_3 scrub vegetation. This is possibly as a result of overgrazing and intensified landuse.

Isotopic Results From Fauna

In total for the late Holocene, isotopic analysis was conducted on the enamel apatite from 21 individual grazers from Rose Cottage Cave, and one grazer from Twyfelpoort. The mean $\delta^{13}C$ values for these grazers are presented in Table 6.6 and plotted out in Fig. 6.3. Analysis of associated bone and dentine collagen from four of the specimens served as a check for possible diagenetic alterations in the enamel apatite. An

Table 6.6 Mean $\delta^{13}C$ apatite values for grazers from the last 2300 years

Site	yr BP ± SD	Level	n	Min. (‰)	Max. (‰)	Mean $\delta^{13}C$ ± SD (‰)	Approximate % C_4 In Diet
RCC	500 ± 50	Mn	6	1.6	3.6	2.7 ± 0.8	100
RCC	680 ± 50	A	6	2.5	3.7	2.9 ± 0.6	100
TFP	1880 ± 50	Lyn	1			1.9	100
RCC	2200 ± 60	A2	8	2.1	3.9	3.0 ± 0.7	100

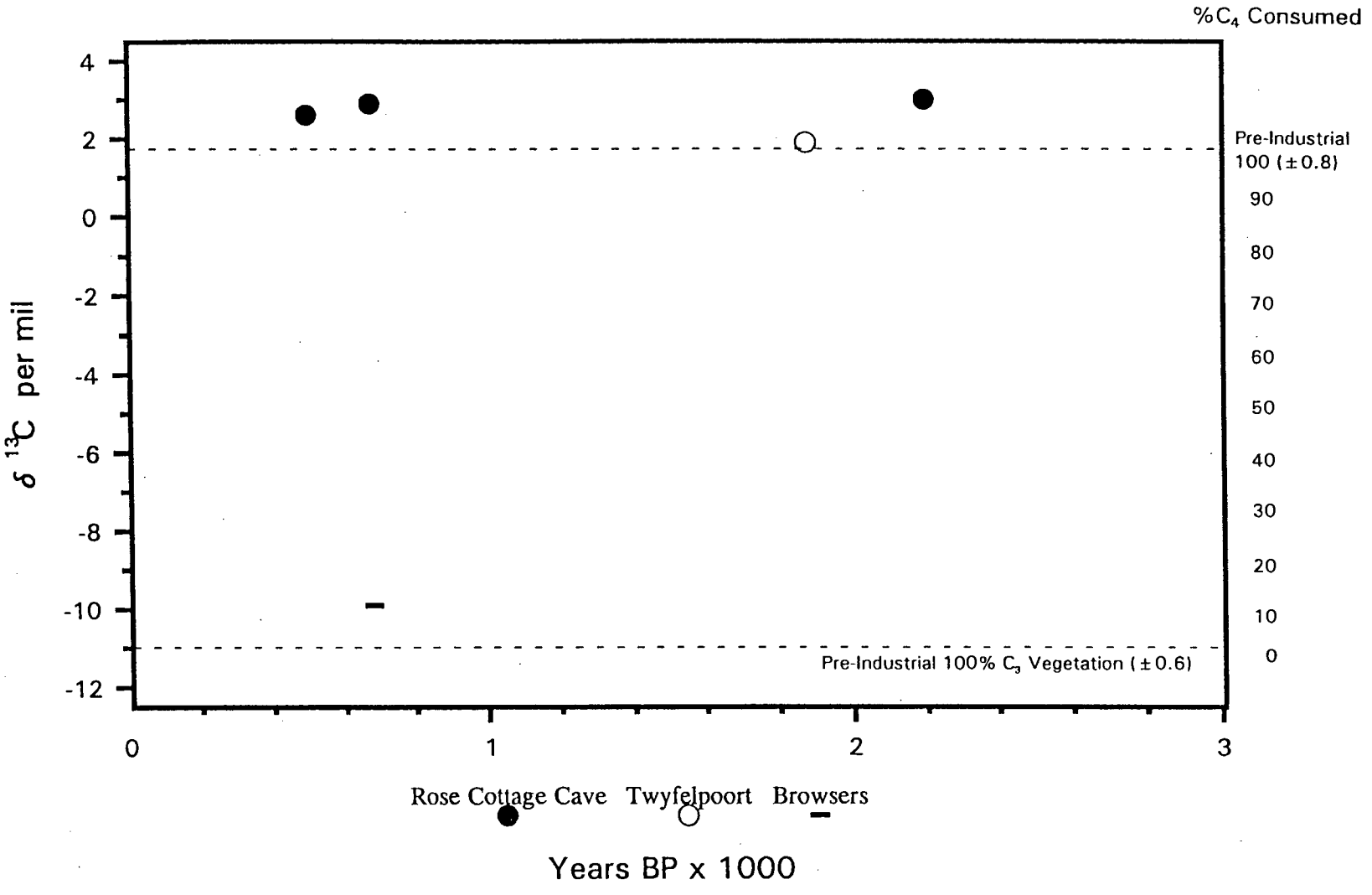

Figure 6.3 Plotted data from Table 6.7: Mean $\delta^{13}C$ values for herbivores for the last 2300 years

approximate 13‰ difference between the enamel apatite of grazers and that of a browser from Rose Cottage Cave at 680±50 BP (see Appendix I section A and Fig. 6.3) provided verification of the results. The raw data for this time period from Rose Cottage Cave and Twyfelpoort are in Appendix I sections A and B.

Grazers from Rose Cottage Cave at 2240±60 BP have a mean $\delta^{13}C$ value of 3.0±0.7‰ (n=8), while the single specimen from Twyfelpoort at 1880±50 BP has a values of 1.9‰. These values demonstrate a 100% C_4 grassland, with $\delta^{13}C$ values consistent with those proposed for modern grazers from the study area. The mean values for grazers from Rose Cottage Cave at 680±50 BP (2.9±0.6‰, n=6) and 500±50 BP (2.7±0.8‰, n=6) indicate a continuation of a pure C_4 grassland.

Isotopic Results From Soil Sediments

Soil samples were taken from varying depths in Donga 1 (n=9) and Unit 1 (n=10), along with sediments from Rose Cottage Cave (n=9), for isotopic analysis of soil organic matter (SOM). Where preserved the macro-organic (MO) remains were also analysed. The data from these analyses are displayed in Table 6.7 and illustrated in Fig. 6.4.

The $\delta^{13}C$ values for SOM from the upper 0.50m of Donga 1

Table 6.7 $\delta^{13}C$ values for soil organic matter (SOM) and macro-organics (MO) from the study area

UCT #	Site	Depth (m)	yr BP ± SD	Level/ Square	$\delta^{13}C_{SOM}$	$\delta^{13}C_{MO}$
5162	RCC	0.10	500 ± 50	Mn/N6	-20.4	
5161	RCC	0.27	680 ± 50	A/L3	-20.2	
5163	RCC	0.42	5970 ± 70	Pt-UP/Q5	-19.9	
5164	RCC	0.48	8160 ± 70	Ja/N5	-22.8	
5165	RCC	0.58	8350 ± 70	Ph/N5	-22.1	
5166	RCC	0.72	ca.8500	Cm/N5	-21.2	
6168	RCC	0.80	8614 ± 38	Ha/N5	-21.4	
5169	RCC	0.90	ca.12 000	DCM/L3	-21.8	
5170	RCC	1.00	12 690 ± 120	DB/L3	-22.6	
5171	Donga 1	0.10			-13.4	
5172	Donga 1	0.20	Post AD 1950		-12.3	
5173	Donga 1	0.30			-13.7	
5174	Donga 1	0.40			-12.6	
5175	Donga 1	0.50			-12.8	-13.2
5176	Donga 1	0.60			-14.4	
5191	Donga 1	0.70	1590 ± 70		-14.4	
5192	Donga 1	0.80			-13.9	
5193	Donga 1	0.90			-15.2	-15.3
5513	Unit 1	0.00			-13.6	-12.5
5514	Unit 1	0.05			-12.5	
5515	Unit 1	0.10			-12.4	
5516	Unit 1	0.15			-12.4	-14.2
5517	Unit 1	0.20			-12.6	
5518	Unit 1	0.25			-13.7	
5519	Unit 1	0.30			-12.4	
5520	Unit 1	0.35			-12.7	
5521	Unit 1	0.40			-13.1	

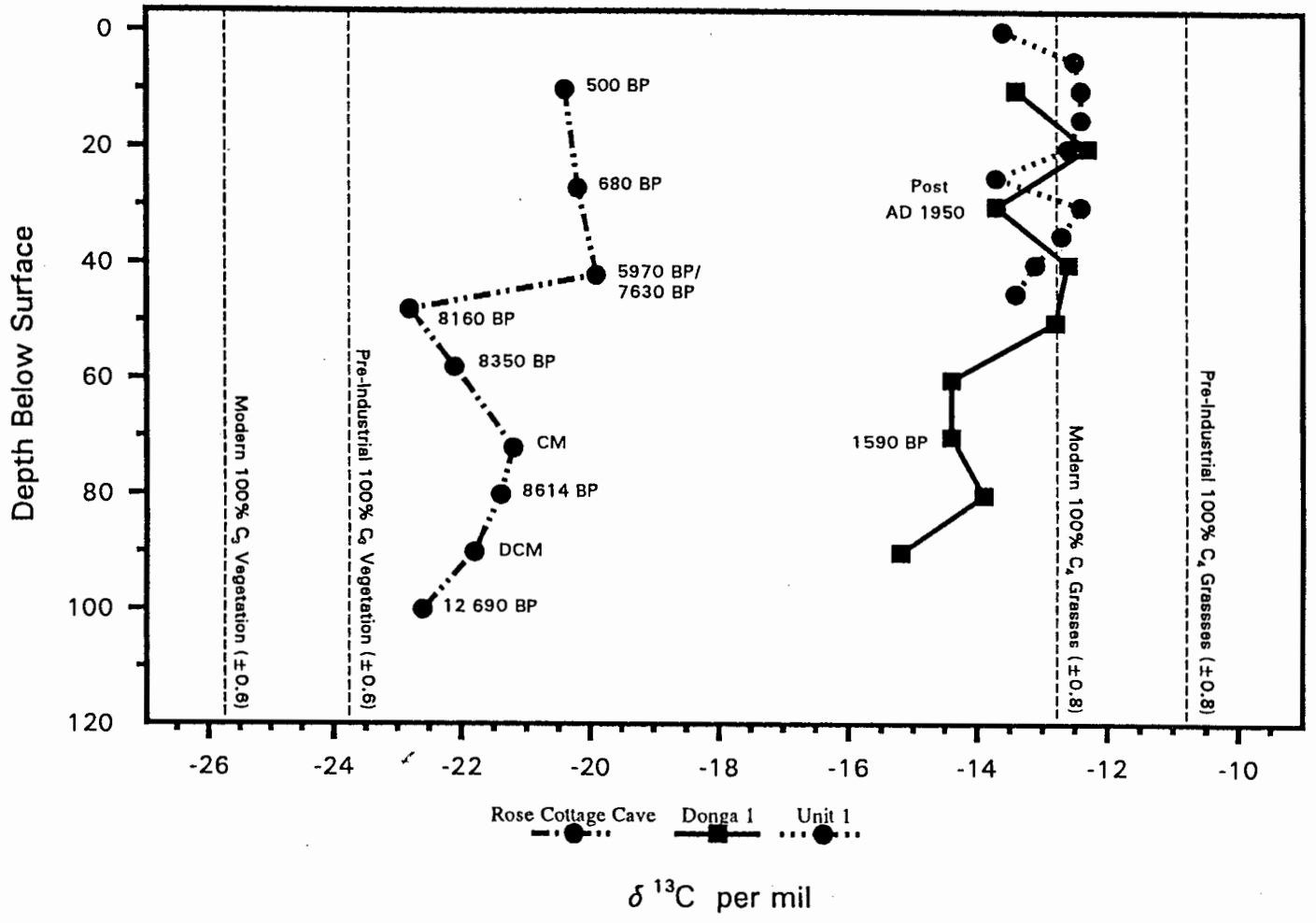

Figure 6.4 Plotted data from Table 6.7: $\delta^{13}C$ values for soil organic matter from the Ladybrand area for the last 2300 years

(n=5) and Unit 1 (n=10) (see Table 6.7) are within the range for modern grasses (mean=-12.7±0.8‰) from the Ladybrand area. Values for analysed MO reflect similar values as the SOM. The upper 0.30m of both sequences are considered to be associated with modern organic accumulations, with a certain portion of recent material filtering down a further 0.10 to 0.20m. Fig. 6.4 illustrates that for Donga 1, below 0.50m, there is a decrease in ^{13}C with increased depth. Four samples from between 0.60 and 0.90m have $\delta^{13}C$ values of -14.4‰, -14.4‰, -13.9‰, and -15.2‰. These specimens are depleted in ^{13}C by 2 to 3‰ in comparison to modern SOM values, and 4 to 5‰ in relation to the estimated value of -10.7‰ for the recent pre-industrial C_4 grassland. It is expected that under stable vegetational conditions, SOM should be enriched in ^{13}C, by 1 to 3‰, with increased depth, due to mircobiotic activity. This being the case, the SOM from between 0.60 and 0.90m (ca. 1600 BP) may reflect a vegetational cover having a $\delta^{13}C$ value of around -16.0 to -18.0‰. The depletion in SOM is supported by the MO value of -15.3‰ at 0.90m. Rootlets tend to show no enrichment or depletion in ^{13}C with depth (Dzurec *et al.* 1985; Skjemstad *et al.* 1990), and therefore, should approximate the vegetation at the time of soil formation. The slight enrichment in ^{13}C of plant roots compared to the value of the above ground portion, may account for the difference between SOM and MO (Cerling *et al.* 1991:297).

In comparison, the SOM analysed from the late Holocene, as well as the terminal Pleistocene to middle Holocene levels, from Rose Cottage Cave is dominated by C_3 vegetation (see Table 6.7 and Fig. 6.4). In relation to the estimated pre-industrial values, C_4 grasses comprised less than 25% of the vegetation deposited in any one level. These values are most likely the result of both cultural and environmental influences. Most of the vegetation brought into the cave through human activity would be C_3 in nature. For example, most edible plants, leaves for bedding, and fire woods are C_3. Environmentally, leaves, along with a small amount of C_4 grasses, were probably washed or blown into the cave. This may suggest a strong C_3 presence, in the form of trees and shrubs, on the slopes around the cave as early as 13 500 years ago.

Interpretation $\delta^{13}C$ Values

The isotopic results of grazers from Rose Cottage Cave and Twyfelpoort indicate the establishment of a 100% C_4 grassland from ca. 2300 BP to the present. Implications are that the general climatic pattern would have been similar to that of today, that is with predominantly summer rainfall and growth temperatures above the 25°c/8°C threshold for C_4 grasses. The pure C_4 diet of both domesticated cow (*Bos taurus*) and migratory grazers at ca. 500 BP suggests that these conditions were in

place on a local and regional scale.

This local climatic pattern is further supported by the $\delta^{13}C$ values of SOM and MO from Donga 1 and Unit 1. Data from all post-1600 BP layers reflect the presence of a 100% C_4 grassland. In addition, the SOM and MO provide no evidence that the intensification of domestic grazers after ca. 500 BP helped to facilitate an increase in scrub vegetation in the study area, as is seen in the Karoo and western Lesotho (Acocks 1988; Hoffman and Cowling 1990; Arbousset and Daumas 1968; Bond *et al.* 1994; Bousman and Scott 1994). However, this may be a reflection of the sampling location, and should not be taken to reflect a lack of grassland deterioration in other areas of the Caledon River Valley.

Change in the predominantly C_4 grassland is registered in both the SOM and MO from Donga 1 ca. 1600 BP. This increase in C_3 vegetation is probably due to climate, as cultural influences on the natural landscape are still minimal. For instance, there is no evidence in the archaeological record for domesticated animals or agricultural activities at this time (Plug and Engela 1992; Wadley 1996). Also, cultural or natural burning of vegetation is not considered to be a cause of the depleted SOM and MO values, as no charcoal is present in the sediments from these horizons (Esterhuysen pers. comm.).

It is unlikely that C_3 scrub would have been naturally supported in the past in the area of Donga 1, as this is an open grassy area on top of the Platberg away from rocky outcrops that could have provide protection for scrubby vegetation (see Fig. 5.4). Based on all these factors combined the depleted $\delta^{13}C$ values for the SOM and MO are mostly likely attributed to an increased abundance of C_3 grasses or wetland flora, rather than an expansion of dry C_3 scrub vegetation. If associated with grasses, this would imply cooler growth temperatures, possibly due to an extension of summer rainfall into autumn months. The lack of comparable isotopic data for grazers dating to ca. 1600 BP prevents assessing whether similar conditions were in place throughout their migratory route. Likewise, a sufficient increase in moisture to cause the area to be waterlogged would result in the growth of forbs and woody C_3 flora.

Site And Regional Comparison Of Palaeoenvironmental Evidence

The lack of well dated sequences has resulted in a poorly documented palaeoenvironmental record for the study area over the last ca. 2300 years. In general, the comparative data for the late Holocene - fauna, sediments, and charcoal - from Rose Cottage Cave, Twyfelpoort and other rock shelters in the western Caledon River Valley, support the stable carbon isotopic

results for a summer rainfall pattern and growth temperatures similar to those of present. The change in vegetation composition ca. 1600 BP reflects a period of climatic fluctuation within this pattern.

Faunal assemblages from Rose Cottage Cave and Twyfelpoort contain a relatively high proportion of grassland species of grazers, suggesting an open grassland environment throughout the late Holocene (Plug and Engela 1992; Wallace 1993). Sediments from the site of Rose Cottage Cave dating to between 1500 - 1000 BP were deposited under moist conditions (Butzer 1984b: 245). For Donga 2, Butzer (1983, 1984b) concluded that the soil horizon dated to ca. 2310 BP was developed in a wet micro-habitat. This wet habitat horizon is considered to be associate with the Donga 1 horizon dated to ca. 1600 BP (Vogel pers. comm.). Further research is required to determine if, and by much how, wetland vegetation from this areas is depleted in ^{13}C in relation to that of dryland, and thus a source contributing to the depleted $\delta^{13}C$ values recorded for Donga 1. Esterhuysen (1996:174-175) provides a tentative climatic interpretation for the last 2000 years based on charcoal analysis. She stresses that amongst other things, dating, vegetation lags, stratigraphy, and the influences of anthropogenic factors, which are typical of this period, render charcoal interpretation problematic. Nevertheless,

indications that conditions were drier than present at ca. 1900 BP, and warm and dry at ca. 680 BP are consistent with the isotopic data.

Within a larger context, the isotopic data for the Caledon River Valley do not support the frequently cited climatic model constructed by Tyson and Lindesay (1992) for the last 2000 years in southern Africa. During their proposed periods of cooler temperatures from ca. 1900 - 1800 BP, ca. 1400 - 1100 BP, and for the "Little Ice Age" from ca. 700 - 150 BP (Tyson and Lindesay 1992:275), the corresponding isotopic data reflects climatic conditions that favour a 100% C_4 grassland. It may be that growth temperatures for the western Caledon River Valley were not sufficiently cool enough to warrant the development of C_3 grasses in the area. If this is the case, further investigation is required to determine if temperatures were low enough to cause an altitudinal expansion of C_3 grasses at sites in western Lesotho. A further period of discrepancy is that of ca. 1750 - 1400 BP, during which Tyson and Lindesay (1992:275) suggest that temperatures were warmer. Isotopic values for SOM indicate an increased abundance of C_3 plants ca. 1600 BP. This has been attributed to an increase in moisture, or cooler growth temperatures either within the established summer rainfall pattern, or as result of an extension of rainfall into the cooler autumn months.

The lack of corresponding climatic episodes should not be taken to mean that the isotopic results are anomalies within a more regional climatic model. Until there is higher resolution dating and data for the Caledon River Valley, as well as for the eastern Free State and Lesotho, it is not possible to determine to what degree, or if, this area fits into the proposed climatic models for southern Africa (Tyson 1986; Tyson and Lindesay 1992).

Summary

Stable carbon isotopic data from grazers and SOM show that a C_4 grassland has been in place in the western Caledon River Valley for ca. 2300 years. This stability is attributed to a summer rainfall pattern and growth temperatures above the C_4 threshold, which are analogous to those of present. Within this general C_4 trend, SOM data provide evidence of an increased abundance of C_3 vegetation in the Ladybrand area at ca. 1600 BP. This has been interpreted as being either an increase in moisture, or lower growth season temperatures, with the possible extension of summer rainfall. Within the areas sampled, cultural alteration of the grassland appears minimal from ca. 2300 BP to present. Neither SOM nor MO provide evidence for an expansion of C_3 scrub, which is known to occur with the intensification of both grazing and agricultural practices.

CHAPTER 7

CONCLUSIONS

The distinct isotopic signature and physical requirements of C_3 and C_4 vegetation have provided the basis for palaeoenvironmental reconstruction in the Caledon River Valley. Although vegetation is seldom directly preserved in deposits, the $^{13}C/^{12}C$ ratio of vegetation is transferred through trophic levels in predictable manner. In this way, the isotopic study of calcified tissues from ungulate grazers and soil organic matter provided a $\delta^{13}C$ index of vegetation change from archaeologically associated samples spanning the last 13 500 years.

Two different kinds of data were selected for this study in order to provide both a local and more regional understanding of palaeoenvironmental change. It was assumed that little or no disturbance took place in the soil profiles sampled from the Ladybrand area, and therefore, that the $\delta^{13}C$ results for soil organic matter reflected the *in situ* C_3/C_4 abundance at the time of soil formation. It was noted, however, that the soil organic matter obtained from Rose Cottage Cave was an inconclusive indicator of palaeoenvironmental conditions, as it was deposited by both cultural and natural agents. On the other hand, the examination of enamel apatite from grazers, which were known

historically to have migrated across the Grassland Biome of KwaZulu-Natal, Lesotho, and the Free State, was seen to exhibit a more regional vegetational pattern.

A discussion of the underlying factors responsible for the vegetational composition of the modern grassland biome was undertaken to provide a better understanding of palaeoenvironmental changes. It became clear, due to the wide ranging migration patterns of the animals, that two different scenarios pertained to the study area. In the low-altitude grassland areas a shift in rainfall pattern would be responsible for altering the C_3/C_4 composition of the grasses, whereas in the high altitude grasslands of Lesotho, fluctuation in temperature was considered to be the primary factor influencing expansion or contraction of C_3 grasses.

In Chapter 6 a modern C_3/C_4 baseline was set up from the analysis of modern material from the eastern Free State and Lesotho. This provided a set of values from which to evaluate changes in $\delta^{13}C$ values obtained from the archaeological material. From the study it became apparent that although, due to present climatic conditions, the modern grassland composition of this region is purely C_4, the isotopic results from grazers and soil organic matter, from in and around the archaeological sites, indicated the presence of C_3 grasses at various times during the terminal Pleistocene

to late Holocene. Part Two of Chapter 6 discussed the presence of these C_3 grasses in terms of an extension of summer rainfall, fluctuation in growth temperatures, and micro-climatic conditions.

The overlap of the terminal Pleistocene to middle Holocene occupational sequences for the western Lesotho sites of Tloutle, Ha Makotoko, and Ntloana Tsoana, with that of the eastern Free State site of Rose Cottage Cave resulted in a relatively high resolution palaeo-environmental sequence. The contemporaneity of isotopic data for grazers also allowed for the identification of possible differences in growth temperatures between the flat grasslands of the eastern Free State and those of the more mountainous regions of western Lesotho. This variation was particularly evident during the terminal Pleistocene and early Holocene. The $\delta^{13}C$ values for both Rose Cottage Cave and Tloutle at ca. 13 400 BP suggested cool growth temperatures, possibly in connection with an extended summer rainfall season. Although there was a general decrease in the abundance of C_3 grasses, cooler temperatures continued to prevailed in western Lesotho sites between ca. 12 100 - 10 000 BP, while at Rose Cottage Cave evidence indicated that a predominantly summer rainfall pattern and growth temperatures consistent with a purely C_4 grassland were in place by ca. 12 000 BP. From ca. 9300 - 8600 BP data from both Rose Cottage Cave and the western Lesotho sites

demonstrated a return to cooler growth temperatures. This was considered to be in association with extended summer rainfall, as a similar depletion in $\delta^{13}C$ values was recorded for grazers from all four sites. The increased abundance of C_4 grasses in the diet of grazers from all of the sites between ca. 8500 - 6000 BP reflected a shift towards a summer rainfall pattern and high growth temperatures. This trend was interrupted by an excursion towards cooler growth temperatures ca. 7600 BP for Rose Cottage Cave, and ca. 7200 BP for Tloutle. In comparison to the Ladybrand area where a 100% C_4 grassland was evident at about 8100 and 6000 BP, it would appear that cooler growth temperatures prevailed in the Roma Valley and the Phuthiatsana-ea-Thaba Bosiu River Basin until ca. 6900 BP, at which time the first evidence for a purely C_4 grassland was found.

Part Three of Chapter 6 focused on the last 2300 years. Isotopic data was derived from the eastern Free State sites of Rose Cottage and Twyfelpoort, as there was not suitable late Holocene material available from the western Lesotho sites. Apart from the noted depletion in $\delta^{13}C$ values for the Ladybrand ca. 1600 BP, modern grassland conditions seemed to have prevailed for much of the last 2300 years. Given the cultural history and environment of the location sampled, the depleted values for the SOM ca. 1600 BP appear to represent an increase in C_3 grasses or wetland flora, rather than an expansion

of dry scrubby C_3 vegetation. This increase has been interpreted as being associated with a period of high precipitation, or with climatic conditions resulting in lower growth temperatures.

The intensification of grazing and greater exploitation of the landscape that accompanied the arrival of Iron Age Farmers and Europeans did not seem to alter the abundance of C_3/C_4 vegetation, as soil organic matter from the later layers provided no evidence for an expansion of C_3 scrub. However, this may be a reflection of the location sampled. Additionally, the findings did not support the theory that the Grassland Biome was a construct of recent human intervention (Acocks 1988; West 1952; White 1978). There was no evidence from soil organic matter to suggest the presence of a forested environment at any time prior to more extensive exploitation of the area by humans. Indeed, the isotopic results from grazers, and faunal assemblages with high proportions of grazing species, suggested that a grassland environment persisted for most of the past 13 500 years.

In this thesis it has been demonstrated that stable carbon isotope analysis of soil organic matter and enamel apatite from grazers offers a reliable means of detecting climate, and possibly human, related changes in the vegetation of past environments. The combined

use of soil sediments and migratory grazers has shown potential for identifying local, as well as regional and altitudinal variation in the isotopic composition vegetation through time. Being able to determine such variations may aid in distinguishing between environmental conditions occurring at different scales. This may shed some light on possible factors influencing the occupation sites in the study area.

From the results of this study it was determined that further research was needed of specific issues. First, the analysis of grasses and forbs from wetlands needs to be undertaken to determine whether the $\delta^{13}C$ values differ significantly from those of dryland grasses. This, for example, may impact on the interpretation of the depleted soil organic values for ca. 1600 BP from Rose Cottage Cave if the wet micro-habitat identified by Butzer (1984b:245) had an affect on the abundance of C_3/C_4 vegetation. Secondly, samples need to be taken from along an altitudinal gradient in the study area to better understand the relationship between the expansion of C_3 grasses and temperature. Thirdly, more information is required on the isotopic variability between species of ungulate herbivores, and possible variations in habitats that could affect interpretation. Lastly, further data needs to be examined from late Holocene sites when it is forthcoming. This will enable the 'filling' of palaeoenvironmental gaps.

APPENDIX I

RAW $\delta^{13}C$ DATA FOR UNGULATE HERBIVORES

Section A: Rose Cottage Cave data for species of grazers and *browsers

UCT #	Species/ Element Analysed	yr BP ± SD	Level/ Square	$\delta^{13}C_{apatite}$ (‰)	$\delta^{13}C_{collagen}$ (‰)	C/N Ratio	$\delta^{13}C_{humates}$ (‰)
5126	*Bos taurus*/ left 3rd molar	500 ± 50	Mn/P2	2.3			
5141	*Connochaetes taurinus*/ right 2nd molar	500 ± 50	Mn/P1	2.4	-5.6	3.0	
5142	*Connochaetes taurinus*/ left 3rd molar	500 ± 50	Mn/N3	1.6			
5145	*Connochaetes gnou*/ right 2nd molar	500 ± 50	Mn/L1	3.0			
5160	*Alcelaphus buselaphus*/ left 3rd molar	500 ± 50	Mn/L1	3.6			
4995	*Equus sp.*/ incisor	500 ± 50	Mn/L1	3.1			
5143	*Connochaetes taurinus*/ left 2nd molar	680 ± 50	A/N3	2.5			
5146	*Connochaetes gnou*/ right 2nd molar	680 ± 50	A/L5	2.5	-7.0		
5127	*Alcelaphus buselaphus*/ left 3rd molar	680 ± 50	A/K5	3.7			
4986	*Damaliscus dorcas*/ left 3rd molar	680 ± 50	A/M1	3.0			

UCT #	Species/ Element Analysed	yr BP ± SD	Level/ Square	$\delta^{13}C_{apatite}$ (‰)	$\delta^{13}C_{collagen}$ (‰)	C/N Ratio	$\delta^{13}C_{humates}$ (‰)
5153A	*Damaliscus dorcas*/ right 2nd molar	680 ± 50	A/N4	2.8			
5124	*Redunca fulvorufula*/ right 2nd molar	680 ± 50	A/P2	3.1			
5121	**Pelea capreolus*/ left 4th pre-molar	680 ± 50	A/N3	-9.9			
5144	*Connochaetes taurinus*/ right molar	2240 ± 60	A2/N4	3.9			
5147	*Connochaetes gnou*/ right 3rd molar	2240 ± 60	A2/P2	3.2			
5148	*Connochaetes gnou*/ right 3rd molar	2240 ± 60	A2/M4	3.8			
5128	*Alcelaphus buselaphus*/ right 3rd molar	2240 ± 60	A2/N3	3.2			
5136	*Damaliscus dorcas*/ left 3rd pre-molar	2240 ± 60	A2/N4	2.1			
4987	*Damaliscus dorcas*/ left 4th pre-molar	2240 ± 60	A2/L4	2.8			
5137	*Damaliscus dorcas*/ right 2nd molar	2240 ± 60	A2/P3	2.8			
4997	*Equus sp.*/ 2nd pre-molar	2240 ± 60	A2/N4	2.7			

UCT #	Species/ Element Analysed	yr BP ± SD	Level/ Square	$\delta^{13}C_{apatite}$ (‰)	$\delta^{13}C_{collagen}$ (‰)	C/N Ratio	$\delta^{13}C_{humates}$ (‰)
5480	*Connochaetes taurinus*/ left 4th pre-molar	5970 ± 70	Pt-UP/M3	2.0			
5150	*Connochaetes gnou*/ right molar	5970 ± 70	Pt-UP/M3	0.8			
5501	*Connochaetes gnou*/ 2nd phalanx	5970 ± 70	Pt-UP/M5		-8.8	2.8	-14.6
5482	*Alcelaphus buselaphus*/ 3rd pre-molar	5970 ± 70	Pt-UP/O3	3.7			
5483	*Alcelaphus buselaphus*/ 2nd molar	5970 ± 70	Pt-UP/P3	3.7			
5129	*Alcelaphus buselaphus*/ left 2nd molar	5970 ± 70	Pt-UP/O4	3.8			
4990	*Damaliscus dorcas*/ right 2nd molar	5970 ± 70	Pt-UP/L3	3.3			
5481	*Redunca fulvorufula*/ left 1st molar	5970 ± 70	Pt-UP/O3	2.4			
5149	*Connochaetes gnou*/ left 3rd molar	7630 ± 70	Pt-LR/Q3	-2.8			
5002	*Equus cf. burchelli*/ 2nd molar	7630 ± 80	Pt-LR/P4	-2.3			
5487	*Connochaetes taurinus*/ tooth fragment	8160 ± 70	Ja/K3	1.7			

UCT #	Species/ Element Analysed	yr BP ± SD	Level/ Square	$\delta^{13}C_{apatite}$ (‰)	$\delta^{13}C_{collagen}$ (‰)	C/N Ratio	$\delta^{13}C_{humates}$ (‰)
5153B	*Connochaetes gnou*/ right 3rd molar	8160 ± 70	Ja/L3	2.6			
5130	*Alcelaphus buselaphus*/ 3rd molar	8160 ± 70	Ja/O3	2.3			
5488	*Alcelaphus buselaphus*/ left 2nd molar	8160 ± 70	Ja/K5	2.1			
5490	*Alcelaphus buselaphus*/ left 2nd molar	8160 ± 70	Ja/J3	2.0			
5140	*Damaliscus dorcas*/ left 3rd molar	8160 ± 70	Ja/Q3	2.2			
4989	*Damaliscus dorcas*/ left 2nd molar	8160 ± 70	Ja/N4	2.5			
5152	*Connochaetes gnou*/ 2nd molar	8350 ± 70	Ph/M5	0.6			
5047	*Alcelaphus buselaphus*/ 2nd molar	8350 ± 70	Ph/O4	-2.0			
5491	*Alcelaphus buselaphus*/ 2nd molar	8350 ± 70	Ph/M4	0.8			
4998	*Damaliscus dorcas*/ left 3rd molar	8350 ± 70	Ph/N3	-0.7			
5139	*Damaliscus dorcas*/ tooth fragment	8350 ± 70	Ph/P4	-2.7			

UCT #	Species/ Element Analysed	yr BP ± SD	Level/ Square	$\delta^{13}C_{apatite}$ (‰)	$\delta^{13}C_{collagen}$ (‰)	C/N Ratio	$\delta^{13}C_{humates}$ (‰)
5125	*Oreotragus oreotragus/ right 3rd molar	8350 ± 70	Ph/M4	-9.7			
5154	Connochaetes gnou/ left 2nd molar	8380 ± 70	JaG/L3	-0.9			
5489	Connochaetes gnou/ right 3rd molar	8380 ± 70	JaG/L3	-0.6			
5485	Connochaetes gnou/ tooth fragment	ca.8500	Cm/?	-1.1			
5048	Alcelaphinae/ 2nd molar	ca.8500	Cm/K4	-1.7			
4991	Damaliscus dorcas/ right 3rd molar	ca.8500	Cm/K3	-1.1			
4993	Equus cf. burchelli/ tooth fragment	ca.8500	Cm/N4	-3.7			
5486	Equus burchelli/ tooth fragment	ca.8500	Cm/N3	-1.2			
5500	*Pelea capreolus/ left 3rd molar	ca.8500	Cm/?	-11.3			
5492	Connochaetes gnou/ 3rd molar	8614 ± 38	Ha/P4	-5.3			
5157	Connochaetes gnou/ left 1st molar	8614 ± 38	HaPit/O4	-2.0			

UCT #	Species/ Element Analysed	yr BP ± SD	Level/ Square	$\delta^{13}C_{apatite}$ (‰)	$\delta^{13}C_{collagen}$ (‰)	C/N Ratio	$\delta^{13}C_{humates}$ (‰)
4996	*Damaliscus dorcas*/ left 4th pre-molar	8614 ± 38	Ha/O4	-10.6			
4994	*Equus cf. burchelli*/ left 3rd pre-molar	8614 ± 38	HaPit/O4	-3.7			
4988	*cf. Damaliscus dorcas*/ tooth fragment	ca.12 000	DCM/J3	2.0			
5498	*Connochaetes gnou*/ left 3rd molar	12 690 ± 120	DB-UP/N3	0.4			
5133	*Alcelaphus buselaphus*/ left 3rd molar	12 690 ± 120	DB-UP/P4	2.3			
5496	*Alcelaphus buselaphus*/ right 3rd molar	12 690 ± 120	DB-UP/O4	1.8			
5495	*Connochaetes gnou*/ molar fragment	13 360 ± 150	DB-LR/M3	-1.7			
5132	*Alcelaphus buselaphus*/ right 3rd molar	13 360 ± 150	DB-LR/O4	-4.6			
5497	*Alcelaphinae*/ right 2nd molar	13 360 ± 150	DB-LR/N3	-1.8			
5499	*Alcelaphinae*/ molar fragment	13 360 ± 150	DB-LR/K5	-0.4			
4999	*Damaliscus dorcas*/ right 1st molar	13 360 ± 150	DB-LR/P4	-1.6			

UCT #	Species/ Element Analysed	yr BP ± SD	Level/ Square	$\delta^{13}C_{apatite}$ (‰)	$\delta^{13}C_{collagen}$ (‰)	C/N Ratio	$\delta^{13}C_{humates}$ (‰)
4992	*Equus cf. burchelli*/ 2nd pre-molar	13 360 ± 150	DB-LR/N3	-1.6			
5494	*Equus cf. burchelli*/ molar fragment	13 360 ± 150	DB-LR/P4	-6.1			

Section B: Twyfelpoort data for species of grazers

UCT #	Species/ Element Analysed	yr BP ± SD	Level	$\delta^{13}C_{apatite}$ (‰)	$\delta^{13}C_{collagen}$ (‰)	C/N Ratio	$\delta^{13}C_{humates}$ (‰)
5434	*Redunca fulvorufula*/ tooth fragment	1880 ± 50	Ly	1.9			
5438	*Damaliscus dorcas*/ phalanx	1880 ± 50	Ly		-9.3	3.0	-15.8

Section C: Tloutle data for species of grazers

UCT #	Species/ Element Analysed	yr BP ± SD	Layer/Area	Square/Unit	$\delta^{13}C_{apatite}$ (‰)
5441	*Damaliscus dorcas*/ molar	715 ± 65	Interior Area	89/58/006	-9.6
5443	*Connochaetes gnou*/ right 3rd pre-molar	715 ± 65	Interior Area	89/58	3.3
5442	*Equus cf. burchelli*/ molar	715 ± 65	Interior Area	88/59/009	-1.2
5444	*Equus sp.*/ molar	715 ± 65	Interior Area	89/57/009	-2.4
5446	*Equus cf. burchelli*/ molar	ca.5000 - 715	BGL	C9/026	2.2
5452	*Redunca arundinum*/ left 3rd molar	6140 ± 100	CSL-UP	E8/084	1.7
5451	*Alcelaphus buselaphus*/ 2nd molar	6140 ± 100	CSL-UP	E9/052	1.8
5455	*Redunca fulvorufula*/ right 3rd molar	6140 ± 100	CSL-UP	D9/133	0.7
5450	*Damaliscus dorcas*/ 1st molar	6910 ± 80	CSL-UP	C10/033	3.8
5449	*Damaliscus dorcas*/ right 2nd molar	6910 ± 80	CSL-UP	D10/033	3.1
5456	*Redunca fulvorufula*/ right 4th pre-molar	6910 ± 80	CSL-UP	D9/140	3.6

UCT #	Species/ Element Analysed	yr BP ± SD	Layer/Area	Square/Unit	$\delta^{13}C_{apatite}$ (‰)
5453	*Equus zebra*/ right 2nd pre-molar	ca.7230 ± 80	CSL-UP	E8/085	-3.8
5454	*Connochaetes gnou*/ right 2nd molar	ca.7230 ± 80	CSL-UP	E7/085	-8.0
5457	*Connochaetes gnou*/ right 2nd molar	7230 ± 80	CSL-LR	E7/124	-7.3
5458	*Equus sp.*/ tooth fragment	ca.9000	BS	E9/068	-4.6
5459	*Damaliscus dorcas*/ 2nd molar	ca.13 400	BC	E10/044	-7.3
5460	*Equus cf. burchelli*/ right 3rd molar	ca.13 400	BC	E9/044	-1.7

Section D: Ha Makotoko data for species of grazers

UCT #	Species/ Element Analysed	yr BP ± SD	Layer	Square/Unit	$\delta^{13}C_{apatite}$ (‰)
5462	*Equus cf. burchelli*/ left 4th pre-molar	8370 ± 80	GWA	K45/013	-1.9
5468	*Equus cf. burchelli*/ left 2nd pre-molar	8370 ± 80	GWA	K44/017	-0.7
5463	*Equus cf. burchelli*/ 1st molar	9290±90	BLOS-UR	K45/034	-3.8
5464	*Connochaetes gnou*/ 1st molar	9290 ± 90	BLOS-LR	K45/034	-10.9
5467	*Equus cf. capensis*/ right 3rd molar	9970 ± 90	BLOS-LR	J45/037	-3.0
5466	*Equus cf. burchelli*/ right 1st molar	9970 ± 90	BLOS-LR	J45/038	-3.0
5465	*Connochaetes gnou*/ left 2nd molar	9970 ± 70	BLOS-UR	J43/037	-0.9

Section E: Ntloana Tsoana data for species of grazers

UCT #	Species/ Element Analysed	yr BP ± SD	Layer/Area	Square/Unit	$\delta^{13}C_{apatite}$ (‰)
5475	*Equus burchelli*/ 3rd incisor	8780 ± 30	MCS	K9/011	-2.7
5470	*Equus burchelli*/ molar	ca.9300-8800	Area L/M14	M14/008	-6.2
5471	*Equus burchelli*/ 3rd incisor	ca.9300-8800	Area L/M14	L14/009	-7.3
5472	*Equus burchelli*/ 2nd molar	10 200 ± 100	Area L/M14	L14/014	-0.1
5473	*Equus burchelli*/ left 3rd molar	10 200 ± 100	Area L/M14	M14/014	-3.0
5479	*Equus burchelli*/ 4th pre-molar	12 110 ± 120	BLOS	K9/032	-10.4

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