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T. Douglas Price *Editor*

Isotopic Proveniencing and Mobility

The Current State of Research

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Cover illustration: Kelly Knudson preparing samples for analysis in the Laboratory for Archaeological Chemistry at the University of Wisconsin-Madison (Photo: T. Douglas Price).

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Preface

Isotopic proveniencing involves the isotopic characterization (O, Sr, and Pb) of tooth enamel or petrous bone. Applications in archaeology involve both humans and animals. Isotopic ratios of these elements vary geographically. As enamel and petrous bone form in early childhood and often do not change, they retain the chemical signature of the place of birth. Comparison with isotopic ratios from the place of burial allows one to distinguish local from non-local individuals. Sulfur isotopes in bone convey information on coastal versus inland provenience and can be useful in studies of adult mobility. The combination of isotopic proveniencing and aDNA has moved archaeological interest in migration and mobility to the fore, but there is very little overview published for either methodology for archaeologists and bioarchaeologists.

I have several things to say about isotopic proveniencing, and I realized that this could best be done in an edited volume with other papers on the subject. This volume should provide state-of-the-art presentation and discussion of procedures, especially what works and what doesn't—what's been learned over the last 30 years that isotopic proveniencing has been underway in archaeology. There is no current consensus on best practice. The contributors have been selected from among the leaders in the field, those with active research and hands-on experience with the approach. Moreover, focus in the volume will be on application, not method, to emphasize to the reader the wide range of questions that can be addressed using isotopic proveniencing. A series of suggestions and recommendations conclude the volume.

Isotopic proveniencing has become an almost standard procedure in the analysis of archaeological burials as a means of distinguishing locals from foreigners. The number of publications has undergone at least a tenfold increase in the last 20 years. At the same time, the field has evolved and new procedures and guidelines have emerged that have not been widely disseminated. New directions emerge that are not always fruitful. This volume aims at state-of-the-art examples and discussion of the new directions and limitations that apply to isotopic proveniencing, including topics such as samples, baselines, isoscapes, distinguishing non-locals, and place of origin.

The hype cycle is perhaps a useful way to characterize the field of isotopic proveniencing (Fenn & Raskino, 2008; Jones & Bösl, 2021). The hype cycle describes the lifespan of an idea or innovation as a series of highs and lows associated with scientific or technological opportunities. This cycle moves from an initial trigger to a peak of expectations, often followed by a trough of disillusionment, then a rising slope of enlightenment and a plateau of productivity. This cycle in archaeology has certainly characterized radiocarbon dating and aDNA research. It would seem that isotopic proveniencing fits this description as well and may be entering a phase of disillusionment at the moment. My own perspective, hopefully demonstrated in this volume, is certainly positive. The field has enormous potential and some problems to be solved.

I would especially like to thank the individuals who have contributed to this volume for their time and input. Bioarchaeology is a rapidly growing field, and the demands on one's time and energy are unlimited relative to what most of us can do. It is the thought and effort of the contributors that gives this volume any consequence that it may have.

I must also thank the staff of Springer, and especially Jelmer Eerkens, the editor of the *Interdisciplinary Contributions to Archaeology* series, and Christi Lue, associate editor for Archaeology & Logic & Cognition. I would also thank the reviewers of the papers and the book. There is no question that your input improved this volume.

Madison, WI, USA T. Douglas Price
November 2022

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Contents

Chapter 1
An Introduction to Isotopic Proveniencing

T. Douglas Price

Abstract This introduction to the volume includes a short history of isotopic proveniencing, a discussion of mass spectrometers as essential instruments in these studies, a brief summary of the major features of the various methods, and an introduction to the papers that follow. It is my hope that this introduction will provide a background that will allow the contributed papers to have more impact.

Keywords History · Mass spectrometers · Isotopic proveniencing · Strontium · Lead · Sulfur · Oxygen

I will do several things in the following pages of this introductory chapter, including a short history of isotopic proveniencing, a discussion of mass spectrometers as essential instruments in these studies, a brief summary of the major features of the various methods, and an introduction to the papers that follow. It is my hope that this introduction will provide a background that will allow the contributed papers to have more impact.

Elements or isotopes that are deposited in the skeleton have the potential to inform on past human activity. Elements or isotopes that are deposited in enamel or the petrous bone and vary geographically have the potential to tell us about childhood conditions. To date, strontium and lead have robust utility for isotopic proveniencing, while oxygen and sulfur are more limited. These four elements and their isotopes are discussed in more detail, following a brief history of isotopic proveniencing and some information on mass spectrometers.

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1.1 A Brief History

Isotopes were commonly under investigation, especially with the advent of commercial mass spectrometers in the 1940s, hastened by the atomic research begun during WWII (Griffiths, 2008). From the 1950s to the present, mass spectrometry has made major strides and innovations are still being made at an enormous pace (Gross, 2021). Access to instrumentation, however, remains a challenge for archaeology. Norman Herz probably led isotope use in archaeology with studies of marble provenience in Italy (e.g., Herz & Dean, 1986). Herz, however, credits Craig and Craig (1972) with the first application of isotopes for provenience in archaeology. Most of the research in archaeology since has focused on diet using carbon and nitrogen, but questions concerning mobility and human proveniencing have taken a larger role in recent years. Strontium, oxygen, lead, and sulfur have been used in these studies.

The relationship between geology and elemental strontium in teeth has been known for some time in dental research (Steadman et al., 1958; Losee et al., 1971). Much of isotopic proveniencing came originally from the geosciences where the geographic variation in strontium isotope ratios was identified and strontium isotopes were used to date rocks. Claire Patterson and George Tipton probably realized that geologically based isotope ratios might be used to determine places of origin for humans. Encouraged by those two famous geoscientists, Jonathan Erickson probably made the connection to tooth enamel that formed in childhood and published the concept of the strontium isotope method (1985). In the same year, Harold Krueger (1985) measured $^{87}Sr/^{86}Sr$ values in fauna and a few humans from the Maya region. Both Ericson and Kruger are now deceased.

Like any new method, adoption was slow and only a few studies appeared in the following decade. Judith Sealy in Cape Town was one light during this period, publishing her thesis (1989) and several papers (1991, 1995) concerned with $^{87}Sr/^{86}Sr$ and diet. At our lab in Madison, post-doc Joe Ezzo suggested to me a trial run for strontium isotope analysis with remains from Grasshopper Pueblo, and our lab was hooked. We involved Clark Johnson and Brian Beard in Geological Science on campus and started getting data. Price et al. published two papers in 1994a, b that employed $^{87}Sr/^{86}Sr$ in specific studies of mobility and several subsequent papers in the next few years. Sillen et al. published a study emphasizing the importance of measuring bioavailable strontium isotope ratios in investigations (1998; Sillen, 2022).

Longinelli (1973) was the first to perform oxygen isotope analysis on bones and teeth deriving from terrestrial mammals. Oxygen isotopes in proveniencing got a start with Willi Dansgaard et al. (1975). Henry Schwarcz et al. (1991) began to work on prehistoric remains, and Chris White et al. (e.g., 1998, 2000, 2002) applied this research in Mesoamerica. Henry Fricke et al. (1995) used the connection to human enamel to compare climate and origin information. One of the early applications involved the Iceman, discovered in 1991 in the Italian Alps (Hoogewerff et al., 2001; Müller et al., 2003).

Lead has been used for years for proveniencing various metal objects of copper and bronze. Its application to prehistoric humans probably began with Gulson (e.g., Gulson et al., 1997). Müller et al. (2003) undertook a multi-isotopic study of the Iceman's tooth enamel, bone, and stomach contents to determine his birthplace, habitat, and range. Their study focused on strontium, lead, and oxygen isotopes to determine his movements. Janet Montgomery's early work, including her PhD thesis, involved both strontium and lead (1999, 2000, 2002, 2010).

The importance of the information from $\delta^{34}S$ measurements was first realized by H.R. Krouse et al. (1987). The first $\delta^{34}S$ values reported for archaeological bone collagen were from a collection of human and animal remains from several South Pacific archaeological sites spanning the last 1000 years in age (Leach et al., 1996). Richards et al. (2001a) demonstrated that sulfur isotopes were able to distinguish inland from coastal regions in archaeological material.

Since 2000, isotopic proveniencing has become almost a standard procedure in the analysis of archaeological burials as a means of distinguishing locals from foreigners. Applications in archaeozoology have also blossomed. The number of publications involving isotopic proveniencing has undergone at least a 20-fold increase in the last 20 years. It has become difficult to keep up with the literature. At the same time, the field has evolved, and new procedures and guidelines have emerged that have not been widely disseminated. This volume aims at state-of-the-art examples and discussion of the new directions and limitations that apply to isotopic proveniencing, including topics such as baselines, isoscapes, and place of origin. But first, an introduction to the instruments that are essential to this research is presented.

1.2 Mass Spectrometers

Because mass spectrometers can resolve atomic weights that differ by less than one atomic mass unit (AMU), they are able to measure isotope ratios for a specific element. Many elements have atoms of more than one atomic weight. Although they are chemically identical, isotopes of the same element have a different number of neutrons—which change the weight but not the chemical properties—in the atomic nucleus. For example, all strontium atoms have 37 protons, which define strontium as a chemical element. Stable strontium atoms can have 47, 49, 50, or 51 neutrons yielding atomic weights (neutrons plus protons) of 84, 86, 87, and 88, respectively. Importantly, one of these, ^{87}Sr, is radiogenic, created by the decay of ^{87}Rb, which has a half-life on the order of 50 billion years. The ratio of ^{87}Sr (roughly 7% in nature) to ^{86}Sr (approximately 10% in nature) varies and has significant archaeological applications.

Mass spectrometers (aka, mass specs) do not use visible light or other wavelengths in the electromagnetic spectrum. These instruments do, however, separate atoms and molecules into a "mass spectrum" according to their weight. They then count the number of particles at a known weight that correspond to a specific element or molecule. Mass specs sort particles according to their weight by putting an

electric charge on the particle and accelerating the particle through an electric or magnetic field (or both).

Table 1.1 (from Wathen et al., 2022) lists some of the similarities and differences among three types of mass spectrometers used in isotopic proveniencing. These instruments are briefly described below.

There are several different types of mass spectrometers. One common type is the quadrupole mass spectrometer in which atoms with an electric charge are accelerated inside of four metal rods to which alternating voltages are applied (Fig. 1.1). If the frequency of the voltage change is just right, then an atom of the desired weight is attracted from one rod to the next as it passes among the rods toward the detector. A lighter atom will be more quickly drawn to one of the electrified rods and expelled; a heavier atom will be too sluggish to respond to the alternating voltage changes. Thus, at a particular voltage and frequency of change, only atoms of a specific mass can successfully traverse the quadrupole device toward the detector; heavier and lighter atoms are thrown out.

Quadrupole mass spectrometers have relatively low resolution and have some problems with interferences. Two elements or compounds with the same atomic weight, for example, CaO^{+1} and Fe^{+1}, both with a mass of 56, cannot be distinguished. Quadrupole mass specs have advantages, however, of being relatively simple and small: these instruments, for example, can be placed on board spacecraft or combined with other analytical instruments such as chromatographs for the identification of molecules. Gulson et al. (2018) have noted problems measuring lead isotopes with quadrupole mass spectrometers and recommend against their use for lead. Others (e.g., Ma et al., 2013) appear to have found solutions to these limitations.

Better resolution of masses and alleviation of the problems with interference can be obtained by using a strong magnet. "Magnetic sector" mass specs accelerate ions into a strong magnetic field, which applies a force pushing the atoms in a direction

Table 1.1 Differences between TIMS, MC-ICP-MS, and LA-MC-ICP-MS analysis (Buzon & Simonetti, 2013; Copeland et al., 2010; Dolphin et al., 2005; Horstwood et al., 2008; Nowell & Horstwood, 2009; Slovak & Paytan, 2011)

	MC-ICP-MS	LA-MC-ICP-MS	TIMS
Sample type	Organic and inorganic	Inorganic	Organic and inorganic
Precision of values	Up to the 5th decimal	Up to the 4th decimal	Up to the 6th decimal
concentration?	Can analyze low	>300 ppm has	Can analyze low
Measurement	concentrations	successful results	concentrations items
Benefits	>50 ppm has	<200 ppm inaccurate	>50 ppm has successful
Issues	successful results	results	results
	One bulk value	Multiple values on the	One bulk value
	Can analyze low	same material	Can analyze low
	concentration	In situ, less time	concentration materials
	materials	consuming	Less susceptible to
	Lacks isobaric	Less destructive	isotopic mass bias
	interferences	Isobaric interferences	Can only produce bulk
	Can only produce bulk	and less precise values	values/destructive
	values/destructive		Time consuming

From Wathen et al. (2022)

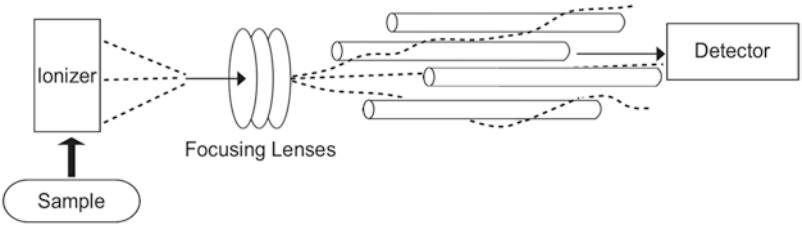

Fig. 1.1 Scheme of a quadrupole mass spectrometer, a beam of atoms of various weights is ionized and focused through four rods to which various voltages are applied. By selecting appropriate DC and high frequency voltages, ions of a specific mass are focused onto the detector, while other masses are rejected

perpendicular to the line of their movement. The magnetic field pushes lower-weight particles to the side more easily than heavy ones, so that the lighter particles are deflected more from their original path. A simple analogy for this mechanism would be a fan blowing perpendicular to a moving stream of golf balls and ping-pong balls. The ping-pong balls will be deflected further from their course than the golf balls. By adjusting the magnetic field, like adjusting the speed of the fan, the deflection can be precisely calibrated so that a particular mass arrives at the detector, while others of a different weight are deflected short or wide of the detector and not counted.

Thus by putting a particle detector at an angle to the original flight path of the atoms, one can focus a particular mass range on the detector by choosing an appropriate magnetic field strength (Fig. 1.2). The strength of the magnetic push is proportional to the speed of the particle, so that varying the electric field that accelerates the particles adds a further degree of control over mass selection. These instruments are generally much larger (and more complicated and costly) than quadrupole instruments. At the same time, magnetic sector mass specs can resolve differences in mass much less than one atomic mass unit (AMU) and can eliminate much of the interference between molecules and atoms with the same weight.

Magnetic sector instruments are further distinguished by their method of sample introduction—how the sample is put into the electromagnetic field. Many instruments today use a plasma, identical to that in optical ICP (inductively coupled plasma) mass spectroscopy (ICP-MS). In the mass spec, the plasma is used to ionize the atoms. Although the plasma emits the characteristic spectra used in ICP-OES (optical emission spectroscopy), this visible light output is not used by the mass spectrometer.

Neither quadrupole nor magnetic-sector machines with just a single ion detector, known as a "collector," have adequate precision (better than 0.01%) for most isotope ratio studies. For isotope ratios, multiple collectors are used so that measurements of the different isotopes can be done at the same time. Multiple collectors are commonly employed with the ICP-MS discussed above ("MC-ICP-MS") and on thermal ionization magnetic mass spectrometers (TIMS). TIMS uses a wire filament on which the purified element of interest is heated to ionize the sample ("thermal ionization"), instead of using plasma.

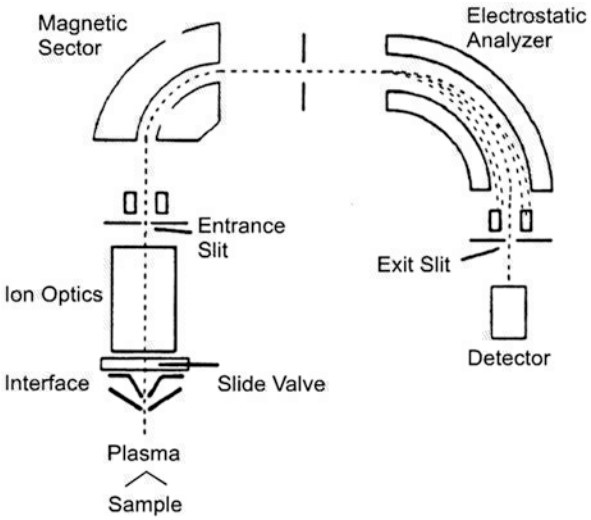

Fig. 1.2 Basic components of ICP-MS. Samples are ionized in the plasma and moved through entrance slit and toward the detector by a magnetic field that separates the atoms by weight. The detector counts the atoms of different weights that arrive

ICP instruments are designed for input of aqueous solutions, but many archaeological samples such as lithics and ceramics are not easily put into solution. Thus a laser is sometimes used on the front-end of the ICP-MS ("laser ablation" or LA-ICP-MS). The laser is focused upon a small spot on the sample and ablates a small part of the material into a gas stream that flows into the plasma. Application of laser ablation to bioapatites is difficult due to isobaric interferences (Lewis, 2015). More on laser ablation can be found in the final chapter Conclusions in the section called Thoughts and Recommendations.

Mass spectrometers usually measure two or more isotopes at one time and calculate their ratio. Standards are measured at the same time in order to confirm the precision of the instrument and allow corrections to be made if needed. The results of the analysis provide the ratio measurements along with several other kinds of information. Table 1.2 shows some typical results from a mass spectrometer, in this case, a list of calculated strontium isotope ratios on human tooth enamel measured on TIMS. The first column in the table gives the sequential lab number for each individual sample. The site is the place from where the sample comes. The corrected $^{87}Sr/^{86}Sr$ is the isotope ratio for strontium in the sample, corrected on the basis of standard reference data collected repeatedly in the instrument. The %standard error is an estimate of error in the isotope ratio. Ratios indicate the number of successful counting episode in 100 tries. Date run tells us when the sample was analyzed. The information on raw 87/86, average of standards, and number of standards measurements is used to correct the final $^{87}Sr/^{86}Sr$ value.

Table 1.2 Typical data from mass spectrometer measurement of strontium isotope ratios in human tooth enamel

Lab number	Site	Corrected $^{87}Sr/^{86}Sr$	%Std error	Ratios	Date run	Raw 87/86	Av of stds	No. of stds
F5025	Xcambo	0.709006	0.0007	88/100	3/23/09	0.709020	0.710264	10
F5026	Xcambo	0.708865	0.0007	89/100	3/23/09	0.708879	0.710264	10
F5027	Xcambo	0.708983	0.0008	86/100	3/23/09	0.708997	0.710264	10
F5028	Xcambo	0.709043	0.0008	87/100	3/23/09	0.709057	0.710264	10
F5029	Xcambo	0.708123	0.0007	87/100	3/23/09	0.708137	0.710264	10

The first three columns provide sample number, site name, and corrected strontium isotope ratio. The remaining columns provide details of the instrument analysis

1.3 Principles and Methods

This section is intended as a brief introduction to the principles and methods for isotopic proveniencing. Isotopic proveniencing of humans involves the isotopic characterization (O, Sr, and/or Pb) of tooth enamel or petrous bone. Isotopic ratios of these elements vary geographically. As enamel and petrous bone form in early childhood and do not change, they retain the chemical signature of the place of birth. Sulfur resides in collagen, not enamel, and thus usually does not inform on childhood status. Sulfur is useful for distinguishing coastal from inland contexts and differences in diet. Carbon isotopes are not an indicator of mobility, but of diet. Carbon is included in some of the discussion here as it is often measured simultaneously with oxygen. Dietary differences are occasionally useful in distinguishing different backgrounds. Focus is on those isotopes and methods that I am familiar with.

Comparison of enamel or petrous bone with certain isotopic ratios from the place of burial allows one to distinguish local from nonlocal individuals. The local isotopic signal of the place of death (or baseline) can be determined in several ways: in human bone from the individuals whose teeth are analyzed, from the bones of humans or archaeological fauna at a site, or from modern fauna, soil, water, or plants in the vicinity. The local geological isotope signals of strontium and lead have been essentially constant over the last several thousand years. The lighter mass of oxygen and sulfur means that there may have been some fractionation (i.e., changes in the isotope ratio) under biological conditions.

Problems with these methods and some solutions are addressed in the last chapter. Samples are becoming more difficult to obtain as curators' field requests from both isotope and aDNA investigators and guidelines for destructive sampling are being introduced or enforced by responsible groups (e.g., Palsdottir et al., 2019).

1.3.1 Strontium Isotopes

Strontium isotopes, reported as the ratio $^{87}Sr/^{86}Sr$, have been predominantly used in the study of human movement in the past. There are several published summaries of the method (Bentley, 2006; Burton & Katzenberg, 2019; Makarewicz & Sealy,

2015; Montgomery, 2010; Price, 2002, 2015; Slovak & Paytan, 2011). Analytical methods are described in detail in a number of publications (e.g., Price & Frei, 2012; Price et al., 1994b; Slovak & Paytan, 2011; Sjögren et al., 2008). Numerous examples of the application of strontium isotope ratios to archaeological questions have been published (e.g., Hedman et al., 2009; Knudson, 2008; Montgomery et al., 2003; Price et al., 2011; Wright, 2005). Some have even referred to a golden age of strontium research (Crowley et al., 2022). However, the method is still experimental and there are a number of problems yet to be resolved.

There are also numerous applications involving the sourcing or proveniencing of other objects and materials such as wood, pottery, stone, textiles, edible plants, or animals. For example, analysis of architectural timbers in Chaco Canyon confirmed a source for the wood some 75–100 km distance (English et al., 2001). In a similar vein, Benson et al. (2003) investigated sources of maize in Chaco Canyon. Several studies of faunal movement have been completed (e.g., Balasse et al., 2002; Hoppe et al., 1999; Sjögren & Price, 2013; Sjögren et al., 2021; Price et al., 2017; Britton et al., 2011). Seasonal variation in animal species behavior has been examined with the use of banded sampling of animal teeth (e.g., Balasse et al., 2002; Meiggs et al., 2018).

The principles of strontium isotope analysis are straightforward. The strontium isotope ratio of $^{87}Sr/^{86}Sr$ varies among different kinds of rocks and sediments. Because ^{87}Sr forms through a radiogenic process as a product of decay from rubidium-87 over time, older rocks with more rubidium have a higher $^{87}Sr/^{86}Sr$ value, while younger rocks with less rubidium are at the opposite end of the range with low ratios (e.g., Montgomery et al., 2006). The half-life for this decay process is 49 billion years, so the abundance of ^{87}Sr does not change on the time scales of human prehistory but can accumulate appreciably on geologic time scales. Regions with old rocks have more ^{87}Sr than regions with relatively younger rocks. Because some rocks and sediments contain higher concentrations of strontium than others, a correction is made using a ratio of the radiogenic ^{87}Sr to non-radiogenic ^{86}Sr. Strontium moves into humans from rocks and sediment through the food chain (Price, 1989; Price et al., 2000; Sillen & Kavanagh, 1982). Strontium is chemically similar to calcium and sometimes substitutes for that element in the formation of enamel and bone.

The amount of ^{87}Sr in nature varies but is ~7% of total strontium and ^{86}Sr is approximately 10%. This ratio normally varies from about 0.700 in very young rocks with low Rb to >0.730 in high Rb rocks that are billions of years old. Most bioarchaeological measurements fall in the range of 0.703–0.723. This ratio generally varies with geology and is not significantly altered by biological processes, so that the $^{87}Sr/^{86}Sr$ ratio in enamel more or less reflects that of the underlying geology and food chain where one was born and raised when the enamel formed. If one moves to a new location with a different geologic setting or is buried in a new place, the enamel isotopes will not change, allowing us to identify the individual as nonlocal.

Dental enamel, especially in teeth only a few millennia old, is relatively robust against diagenetic alteration. Kamenov et al. (2018) recognized three levels of

preservation in archaeological teeth: pristine, slightly altered, and highly altered. Only highly altered teeth showed evidence of alteration in strontium isotopes.

Originally we collected the permanent first molar both for consistency and the fact that the enamel of this tooth forms during gestation and very early childhood. However, over time we have shifted to premolars as the tooth of choice for sampling since this tooth forms after breastfeeding is largely completed, has fewer diagnostic traits of interest for dental studies, and is more common. There are more premolars (eight) than first molars (four). We still sample molars if additional material is needed for dating and/or aDNA study. The significance of third molars, which form later than other teeth (if at all), is discussed further in this volume and in the concluding chapter.

Preparation involves lightly abrading the surface of a single cusp using a dental drill to remove surficial dirt and calculus and also the outermost enamel due to the possibility of contamination by diffusion (Fig. 1.3). This cusp is then cut from the tooth with a crosscut blade and any remaining dentine removed with a drill. If a clean cusp is not available, a small chip is removed from the side of the tooth, or 5–10 milligrams of powder is drilled from the enamel. It is also clear that less destructive methods will be required in the future.

It is the case with cremations, especially in high temperature fires, that enamel tends to explode and is difficult to recover. In such cases, the petrous bone appears to be a good substitute. The petrous bone is one of the densest in the body and forms during early childhood. Its inner periosteal layer is formed in utero and does not undergo remodeling after the age of two (Jørkov et al., 2009). This bone is found in the otic cavity of the skull and usually survives cremation fires. A study of Iron Age cremations in Ljubljana, Slovenia, utilized the petrous bone for samples with good results (Škvor Jernejčič & Price, 2020). Snoeck et al. (2015) have suggested the use of calcined bone from cremations and argue that calcined bone is more resistant to post-mortem exchange than tooth enamel and that in vivo strontium isotopic ratios are retained in the calcined bone. That may well be the case, but calcined bone in most cases does not contain a childhood signal and is less useful for that reason.

Samples of enamel or petrous bone powder weighing 2–5 mg are dissolved in five-molar nitric acid. The strontium fraction is purified using ion-specific resin and eluted with nitric acid followed by water. This solution is loaded onto a titanium

Fig. 1.3 Sampling tooth enamel. The first step is to lightly grind the surface of the enamel to remove potential contamination

filament for placement in the instrument. Isotopic compositions are obtained on the strontium fraction using a thermal ionization mass spectrometer (TIMS) or MC-ICP-MS.

Some investigators are using ICP-MS instruments with multi-collectors to measure strontium isotope ratios. Laser ablation is also sometimes used as a means of sample introduction, although nebulization remains a standard for this instrument. There was debate for some years about the precision of MC-ICP-MS, but these problems appear to have been resolved and more research groups are using ICP instrumentation (e.g., Le Roux et al., 2014; Wall et al., 2016).

$^{87}Sr/^{86}Sr$ ratios are corrected for mass fractionation using an exponential mass fractionation law. $^{87}Sr/^{86}Sr$ ratios are reported relative to a value of 0.710250 for the NIST 987 standard (e.g., if the $^{87}Sr/^{86}Sr$ ratios for the standards analyzed with the samples average 0.710260, a value of 0.000010 is subtracted from the ratio for each sample).

An essential question concerns the local strontium isotope signal for the area in which the burial is located. In actual fact, levels of strontium isotopes in human tissue may vary from local geology for various reasons (Price et al., 2002), and because of this, it is necessary to measure "bioavailable" levels of $^{87}Sr/^{86}Sr$ to determine local strontium isotope ratios. The local bioavailable isotopic signal of the place of burial can be determined in several ways: from the human bone from the individuals whose teeth are analyzed, from the bones of other humans or archaeological fauna at the site, or from modern fauna or plants in the vicinity. The term proxy is often applied to the materials used as an alternate measure of bioavailable $^{87}Sr/^{86}Sr$, that is, an alternate to the original foods that the individual ate. The use of the bone for comparison assumes that either diet or diagenesis has brought bone strontium isotope levels to the local value. Surface water is not always a reliable source of bioavailable strontium, and water usually does not contribute much strontium to human tissue. This baseline information on isotope values across an area needs to be obtained in order to make useful and reliable statements about the origins of the human remains under study (Price & Frei, 2012; Price et al., 2002).

There are a number of problems with strontium isotope analysis, especially the definition of local, and the measurement of bioavailable $^{87}Sr/^{86}Sr$. These issues are considered in more detail in the concluding chapter.

1.3.2 Lead Isotopes

Lead is the element with the heaviest isotope (208) in nature. Lead, in addition to non-radiogenic ^{204}Pb, has not one, but three, radiogenic isotopes: ^{206}Pb, ^{207}Pb, and ^{208}Pb. Measurable differences in lead isotopic compositions are caused by the differential radioactive decay of ^{238}U ($t_{1/2} = 4.5 \times 109$ years), ^{235}U ($t_{1/2} = 0.70 \times 109$ years), and ^{232}Th ($t_{1/2} = 1.4 \times 1010$ years) to form ^{206}Pb, ^{207}Pb, and ^{208}Pb, respectively (Faure & Mensing, 2004). The relative abundances of these isotopes in nature are $^{204}Pb = 1.4\%$, $^{206}Pb = 24.1\%$, $^{207}Pb = 22.1\%$, and $^{208}Pb = 52.4\%$.

The level of lead in the Earth's crust is about 20 mg/kg. Lead in the environment may derive from either natural or anthropogenic sources. Natural sources of atmospheric lead include geological weathering and volcanic emissions and have been estimated at 19,000 tons/year, compared to an estimate of 126,000 tons/year emitted to the air from mining, smelting, and consumption of over three million tons of lead per year in the industrial era (Ursfnyovfi & Hladfkovfi, 2000). Evans et al. (2018) argue that the Pb isotope composition of the silicate (natural) and sulfide (anthropogenic) Pb from soil displays a crustal average ^{238} U/^{204}Pb (μ) value of ca. 9.7. Pb from ore displays a much wider range of evolutionary pathways. These characteristics are transferred into tooth enamel, making it possible to characterize human Pb exposure in terms of the primary source of ingested Pb (Evans et al., 2018).

Summaries of lead isotope studies in bioarchaeology are provided in Sharpe et al. (2019) and Kamenov and Gulson (2014). The chapter in this volume by Kamenov and Krigbaum provides an excellent introduction to lead isotopes in archaeology. Lead (Pb) behaves remarkably like strontium in provenience studies. Both elements substitute for calcium in hydroxyapatite in the skeletal tissue, are transferred with negligible fractionation from soils into plants and through successive trophic levels, and are found as trace constituents in plants and animals. Virtually the entire body burden of both elements is deposited in the skeleton. Bone concentrations of both elements are proportionate to dietary (and hence environmental) abundances, and both elements undergo trophic-level biopurification. Most importantly, both elements have stable and radiogenic isotopes, such that the ratios of these isotopes depend upon the local geology and are thus geographically variable. In contrast to strontium, there is very little lead in seawater or marine foods. Unlike strontium and oxygen isotopes, lead isotope ratios in living organisms are not influenced by coastal ocean spray.

Normally the relative abundance of the different isotopes of an element varies little across the surface of the Earth, but that is not the case with lead. Stable lead isotopes, and their ratios, vary locally according to both the geologic age of the terrain and the amounts of the parent isotopes of uranium and thorium. Importantly, lead isotope ratios, like strontium isotope ratios, are not changed by biological or other low-temperature chemical or physical processes. The ratios present in dental enamel reflect those of dietary intake and, in the New World prior to the modern industrial era, that of the local geology. The four stable isotopes of lead provide a large number of potentially useful ratios, but the values of ^{206}Pb/^{204}Pb, ^{207}Pb/^{204}Pb, ^{208}Pb /^{204}Pb, ^{207}Pb /^{206}Pb, and ^{208}Pb /^{206}Pb are commonly used in isotopic provenience studies. The process of lead isotopic testing is similar to that of strontium, and the two can be measured from the same prepared sample (Sharpe et al., 2019). Methods for sample preparation and analysis are well established in geology (e.g., Baker et al., 2004).

Although there is evidence for diagenesis of lead in the buried bone, this contamination does not appear to be present in tooth enamel in most cases (Gulson & Gillings, 1997; Montgomery et al., 1999; Waldron, 1973). Kamenov et al. (2018) recommend that concentrations of a series of trace elements (Pb, V, Mn, Fe, REE, U, and Th) be measured before a sample can be declared fit for analysis.

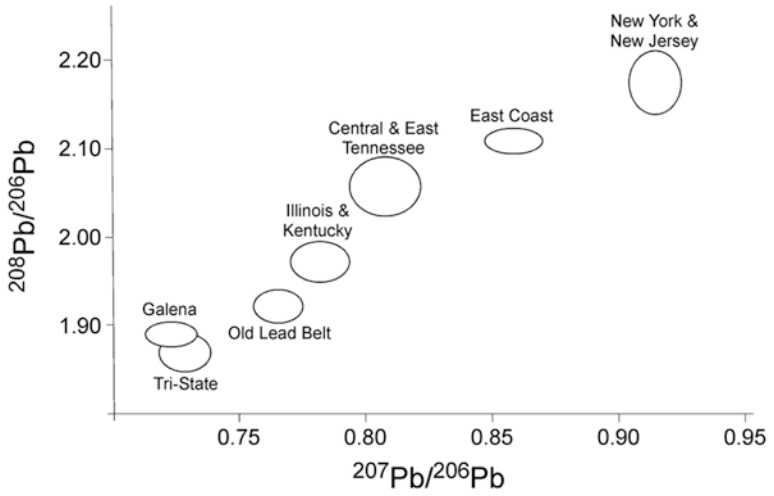

Fig. 1.4 Two isotope ratios of lead, $^{207}Pb/^{206}Pb$ and $^{208}Pb/^{206}Pb$, distinguish sources of lead in the Eastern United States. (Diagram from Augustine, 2002)

Because of their variability, lead isotopes have had a successful history in archaeometric research for artifact provenience studies. Figure 1.4 shows the variation in two lead isotope ratios ($^{207}Pb/^{206}Pb$ and $^{208}Pb/^{206}Pb$) in sources of lead in Eastern North America. Blichert-Toft et al. (2016) have provided new information on lead isotope variation in Europe that may be useful for human proveniencing. Samuelsen and Potra (2020) suggest using prehistoric animal tooth enamel to obtain bioavailable values. Because of their variability, stable lead isotopes have been used successfully in archaeometric research for artifact provenience studies, including the analysis of metals, paints, and pigments, and more recently to identify human movement in the past. Lead and its isotopes have been measured in soils across Europe (Reimann et al., 2012), but modern contamination remains a problem in establishing baseline information.

Lead isotope analysis was initially applied to lead-rich artifacts, such as those made of copper-, bronze-, and lead-bearing ores, to study provenience questions. This method was later used with other artifact classes such as ceramics. Lead isotopes have also been used as a tracer in past human mobility studies (e.g., Augustine, 2002; Budd et al., 1998, 2000; Carlson, 1996; Corruccini et al., 1987; Gulson & Gillings, 1997; Molleson, 1988; Montgomery et al., 1999; Reinhard & Ghazi, 1992; Sharpe et al., 2016). Giovas et al. (2016) have used lead isotopes to look at animal introduction to the Caribbean Islands.

Lead is measured in ultraclean lab facilities. Pieces of tooth enamel or bone weighing between 5 and 20 mg are first bathed in 1 ml of 1 M HNO_3 for 2 min to remove surface contamination. After washing multiple times with ultrapure water, the pieces are then dissolved in 1 ml of a 5:1 mixture of 16 M HNO_3 (Seastar Baseline™ quality) and 30% H_2O_2 Savillex™ in a 7 ml round bottom Teflon vial.

One-fifth of the solution is pipetted off into a new vial and spiked with an adequate amount of a ^{204}Pb-enriched tracer solution for determining the Pb concentrations via an isotope dilution (ID) method. Three-fifths of the solution is used for the measurement of the respective Pb isotope values (IC). Both Pb "ID" and "IC" aliquots of the respective samples are carefully dried on a hot plate not exceeding 80 °C. Ion chromatographic separation of Pb was achieved over miniaturized extraction columns fabricated from 200 µl pipette tips with a fitted frit in the bottom and top of ~200 µl 100–200 mesh AG 1 × 8 anion resin (BioRad™), sandwiched in between the two frits. The HCl-HBr-based extraction schematics used acid volumes adjusted to the miniaturized extraction columns. Procedural blanks remained below 60 pg of Pb, an amount which is insignificant relative to the several nanogram amounts of sample processed for the mass spectrometric analyses. Pb ID and IC fractions were loaded on outgassed 99.98% single rhenium filaments in a mixture of 2 µl silica gel (Gerstenberger & Hasse, 1997) and 2 µl 1 M H_3PO_4. Pb isotopes were statically measured at temperatures between 1020 and 1150 °C on a TIMS. Fractionation of Pb during mass spectrometric analysis was monitored by repeated analysis of the NBS 981 standard (Todt et al., 1996) and amounted to 0.105 ± 0.008%/amu (2σ, $N = 5$).

There are several problems with lead isotope analysis, including the low concentrations of this element in dental enamel, the poorly known geographic distribution of most lead isotope values prior to modern contamination, and the solubility of lead resulting in the potential contamination of modern and ancient samples, particularly from the combustion products of leaded gasoline (e.g., Albarède et al., 2011). Bioavailable levels of lead isotopes are harder to determine because of the ubiquity of modern pollution. Evans et al. (2018) demonstrate that different geological processes control the Pb isotope composition of the silicate (natural) and sulfide (anthropogenic) Pb sources making it possible to characterize human Pb exposure in terms of the primary source of ingested Pb. The ubiquity of pollutants also means that an ultraclean lab is often necessary to avoid contamination issues. These factors make measurement and interpretation of ratios of lead isotopes considerably more complex than for strontium isotopes.

1.3.3 Oxygen Isotopes

Oxygen isotopes have been widely used as a proxy for temperature in many climate and environmental studies and vary geographically in surface water and rainfall (Dansgaard, 1964). Oxygen isotopes in archaeology are used primarily to study ancient environments and to examine past human mobility (Lightfoot & O'Connell, 2016; Pederzani & Britton, 2019). Oxygen isotopes in ancient human skeletal remains are found in both tooth enamel and bone. Oxygen is incorporated into dental enamel during the early life of an individual and remains largely unchanged through adulthood. Samples for the analysis of human skeletal remains are normally taken from dental enamel due to better preservation and resistance to diagenesis.

Oxygen isotopes are also present in bone apatite and are exchanged through the life of the individual during bone turnover, thus reflecting place of residence in the later years of life. Thus, oxygen isotopes have the potential to be used to investigate human mobility and provenience (Bowen et al., 2005). Carbon isotopes (^{13}C, ^{12}C) are frequently measured simultaneously with oxygen on mass specs equipped with a Kiel device. The use of both isotopes of hydrogen in bone collagen has also been suggested (O'Brien & Wooller, 2007), but this application remains largely experimental.

Oxygen has three isotopes—^{16}O (99.762%), ^{17}O (0.038%), and ^{18}O (0.2%)—all of which are stable and non-radiogenic. As with carbon and nitrogen, isotope measurements of oxygen are always reported as a ratio of one isotope to another, lighter, and more abundant cousin. Oxygen isotopes are commonly reported as the per mil difference (‰ or parts per thousand) in the ratio of ^{18}O to ^{16}O between a sample and a standard. This value is designated as $\delta^{18}O$. This value can be measured in either carbonate $(CO_3)^{-4}$ or phosphate $(PO_4)^{-3}$ ions of apatite in the tooth and bone. Phosphate and carbonate often produce comparable results for oxygen isotope ratios (Sponheimer & Lee-Thorp, 1999) (Fig. 1.5).

The oxygen isotope ratio in the human skeleton reflects that of body water, and ultimately of drinking water (Kohn et al., 1996; Kohn et al., 1998; Luz et al., 1984; Luz & Kolodny, 1985), which in turn predominantly consists of local rainfall. Oxygen isotope ratios vary geographically and seasonally in surface water and

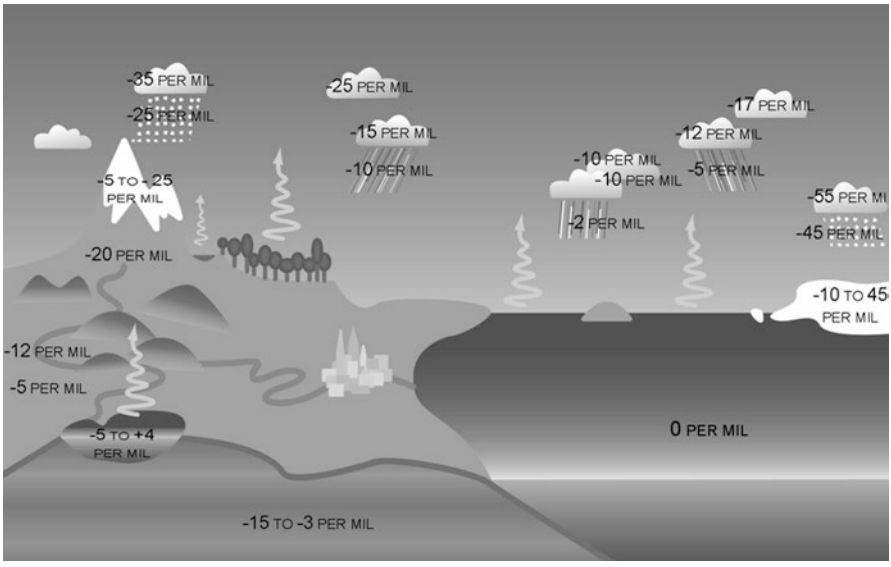

Fig. 1.5 The oxygen isotope ratio measured by $\delta^{18}O$ varies with temperature, latitude, and elevation. Depending on atmospheric temperature, ^{16}O evaporates faster than ^{18}O from the ocean's surface. As rain clouds move inland or toward cooler areas, the heavier isotope (^{18}O) precipitates preferentially, and rain clouds become progressively depleted in ^{18}O as they move inland. $\delta^{18}O$ provides a proxy for atmospheric temperature

rainfall. Isotopes in rainfall are greatly affected by enrichment or depletion of the heavy isotope (^{18}O) relative to the lighter ^{16}O in water due to evaporation and precipitation (e.g., Dansgaard, 1964). Major geographic factors affecting rainfall isotope ratios then are latitude, elevation, amount of precipitation, and distance from source (e.g., an ocean). Rainwater (H_2O) contains five stable isotopes, including ^{16}O and ^{18}O. Molecules of $H_2{}^{18}O$ have a greater mass than $H_2{}^{16}O$ and require more energy to evaporate and to stay in the atmosphere. As moisture moves over land, the first precipitation contains more of the heavy isotope and as clouds move inland, to cooler regions, and to higher elevations, the rain becomes more depleted in the heavier isotope—lower in $\delta^{18}O$ (Bowen & Wilkinson, 2002). Thus, oxygen isotope ratios in teeth vary geographically and have potential to provide information on past human movement by comparing place of tooth formation (childhood) and place of death (Bowen & Revenaugh, 2003).

Oxygen isotopes in ancient human skeletal remains have been analyzed from both the tooth enamel and bone (France & Owsley, 2015). Oxygen is incorporated into dental enamel during the early life of an individual and should remain unchanged in that enamel through adulthood, although some minor changes have been observed (Price et al., 2019). $\delta^{18}O$ can be measured either from isolated carbonate $(CO_3)^{-2}$ or phosphate $(PO_4)^{-3}$ components in apatite in the tooth and bone. The standards used are commonly VSMOW (Vienna Standard Mean Ocean Water) for phosphate oxygen and water (and sometimes carbonate oxygen) and VPDB (Vienna PeeDee Belemnite) for carbonate oxygen (O'Neil, 1986). Samples are typically ground or drilled to powder for analysis; less sample is needed for analysis of the carbonate component, preparation is less demanding, and results between laboratories are more comparable (e.g., Bryant & Froelich, 1995; Sponheimer & Lee-Thorp, 1999). However, there are significant fractionation differences between the carbonate and phosphate sites in apatite (Aufort et al., 2017). The phosphate component is less labile and may preserve original compositions more faithfully, although this issue is subject to debate. There is also substantial discussion of appropriate preparation methods for both carbonate and phosphate $\delta^{18}O$ analyses (Vennemann et al., 2002; Grimes & Pellegrini, 2013; Koch et al., 1997; Pellegrini & Snoeck, 2016). In fact, no pretreatment may be the best alternative.

The $\delta^{18}O$ values for carbonate and phosphate oxygen in enamel vs. $\delta^{18}O$ of drinking water can be estimated assuming equilibration at body temperature and converting for different standards (VSMOW vs. VPDB). Chenery et al. (2012) compared $\delta^{18}O$ values of phosphate and carbonate for 51 archeological samples and derived a relationship between the $\delta^{18}O_{VSMOW}$ value of drinking water (DW) and $\delta^{18}O_{VSMOW}$ in enamel carbonate (EC) as $\delta^{18}O(EC) = (\delta^{18}O(DW) + 48.63)/1.59$. Measurements reported relative to the VPDB can be converted to the VSMOW scale: $\delta^{18}O_{VPDB} = (0.97001 \times \delta^{18}O_{VSMOW}) - 29.99‰$ (Kim et al., 2015). Thus, as an example, a drinking water $\delta^{18}O_{VSMOW}$ value of $-6.0‰$ yields an estimated enamel carbonate $\delta^{18}O(EC)_{VSMOW}$ value of approximately 26.8‰. The best formula for correlating water to tooth enamel oxygen levels is still debated (Iacumin et al., 1996; Pollard et al., 2011; Pryor et al., 2014).

Oxygen is measured in either carbonate or phosphate in human tooth enamel. As tooth enamel is relatively resistant to diagenetic alteration, carbonate analysis of tooth enamel bioapatite is generally considered valid, while carbonate analysis on the bone should be avoided as it is subject to diagenetic alteration. Phosphate should be less susceptible to diagenesis but carbonate analysis is simpler, cheaper, and quicker. $\delta^{18}O$ and $\delta^{13}C$ of tooth enamel carbonate are often measured simultaneously on the carbonate fraction of dental enamel using an automated carbonate preparation device (KIEL-III) coupled with a gas-ratio mass spectrometer. Powdered samples are reacted with dehydrated phosphoric acid under vacuum at 70 °C in the presence of silver foil. The isotope ratio measurement is calibrated based on repeated measurements of NBS-19 and NBS-18, and precision is ±0.1‰ for $\delta^{18}O$ and ±0.06‰ for $\delta^{13}C$.

Lightfoot and O'Connell (2016) conclude that identifying individuals' homelands on the basis of oxygen isotope analysis alone is not possible for most of Europe. There remain substantial problems using $\delta^{18}O$ for geographic provenienc-ing. Concerns with and limits of oxygen isotope analysis are discussed in both Freiwald's chapter and the concluding chapter of this volume. Freiwald in this volume considers the use of oxygen isotopes in Mesoamerica and points out a number of problems and questionable areas in such studies.

1.3.4 Sulfur Isotopes

There are four stable isotopes of sulfur: ^{32}S (95.02%), ^{33}S (0.75%), ^{34}S (4.21%), and ^{36}S (0.02%) (Trust & Fry, 1992). The ratio between the two most abundant isotopes, ^{32}S and ^{34}S, is defined as the $\delta^{34}S$ value, measured relative to the Vienna CDT standard (Coplen & Krouse, 1998). Sulfur is abundant with two significant isotopically uniform reservoirs: the Earth's metallic core, with a $\delta^{34}S$ value near 0‰, and oceanic sulfate ($S0_4^-$), with a $\delta^{34}S$ value near +20‰. Thus, there are significant differences among the $\delta^{34}S$ values of plant and animal specimens from freshwater and marine ecosystems (Richards et al., 2001a). Sea spray can carry sulfur particles inland and cause coastal soil $\delta^{34}S$ values to be similar to those of the ocean (Wadleigh et al., 1994). Quality control criteria for sulfur isotopes in bone collagen have been suggested by Nehlich and Richards (2009) allowing standardization among labs. A number of studies have shown the potential of sulfur isotope analysis in palaeodietary research.

Sulfur isotope analysis has also been used for the reconstruction of mobility patterns in the past (Drucker et al., 2011; Nehlich, 2015; Nehlich et al., 2012; Vika, 2009; Wißing et al., 2019), employing the distinct sulfur isotope ratios of inland vs. coastal regions. There are limits, however. As sulfur occurs in collagen, not enamel, there is no childhood signal available so that values of $\delta^{34}S$ reflect the later years of life. There is also a dearth of reliable information on ancient bioavailable levels of $\delta^{34}S$—modern values have been contaminated by pollutants such as acid rain.

Richards et al. (2003) suggest that archaeological finds might provide baseline data. Richards (this volume) argues for the utility of sulfur isotopes in mobility studies.

Description of the measurement of sulfur isotopes in organic materials is provided by Nehlich (2015). Measurement is undertaken by the direct combustion of the collagen and the measurement of the resultant SO and SO_2 gases using continuous flow isotope ratio mass spectrometry (CF-IRMS) (Giesemann et al., 1994; Baublys et al., 2004; Yun et al., 2004). Ratios of heavy to light isotopes need to be statistically corrected against the possible occurrence of heavy oxygen isotopes within the sulfuric gases because they might mask the actual number of heavy and light sulfur isotopes (Fry et al., 2002; Coleman, 2004). Mayer (2009) suggests that both the analytical method and the resulting isotopic compositions for the international standards should be made explicit.

1.4 Chapters in This Volume

1.4.1 T. Douglas Price: Introduction

This introductory chapter has provided a short history of isotopic proveniencing, a discussion of mass spectrometers, and a brief summary of the various methods. Now, an introduction to the chapters that follow in the volume:

1.4.2 Kelly Knudson, Christina M. Torres, and William Pestle: Isotopic Analyses in the Andes: From the Macro- to Microscale

Knudson et al. offer a detailed look at isotopic proveniencing in the Andes, a region of enormous environmental, geological, and altitudinal variability. They provide an overview of both regional variations in the area along with specific site studies. In addition, they provide substantial insight on individual life histories in regard to some of the sacrificed frozen mummies.

1.4.3 Carolyn Freiwald: Oxygen Isotope Values in the Maya Region

Carolyn focuses on the application of oxygen isotopes in Mesoamerica and inherent problems with that system. First, she considers the use of different samples and tissue types and shows how different treatment protocols may affect comparability of

samples. She then explores the utility of oxygen isoscape models in Mesoamerica, which show mixed results in their potential to predict human isotopic variation.

1.4.4 Rick Schulting, John Pouncett, Christophe Snoeck, Hannah F. James, and Warren Bailie: Common Ground: Investigating Land Use and Community Through Strontium Isotope Analysis of Bronze Age Cremations from Dunragit, Southwest Scotland

Schulting et al. use strontium isotopes to examine landscape use by the local Bronze Age community. They report strontium isotope and concentration values for 24 cremations from Dunragit, Southwest Scotland, together with measurements on 23 modern plants to refine the available regional isoscape. Their focus is more on the use of the landscape than on human mobility, a growing trend in isotopic proveniencing studies.

1.4.5 Anne Waterman: Traveling Up Hill and Down Dale: Using Isotopic Studies of Human and Animal Mobility in Chalcolithic Portugal to Investigate Intraregional Patterns of Social and Economic Relationships in Late Prehistory

Anne focuses on strontium isotope data from neighboring Late Prehistoric sites in Southwestern Portugal, examining both humans and animals to find evidence of small-scale, short-distance migration and adjacent-site travel with the goal of understanding intraregional, cultural, and economic integration.

1.4.6 T. Douglas Price and Dusan Boriç: The Transition to Agriculture

This chapter concerns one of the major questions in archaeology and considers aspects of mobility and sedentism among hunters and farmers. Using two case studies—the Danube Gorges and Southern Scandinavia, dated human remains and strontium isotopes—it is possible to suggest from the timescale of a lifetime that early farmers probably moved more than hunters.