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Michael Sperazza
The University of Montana

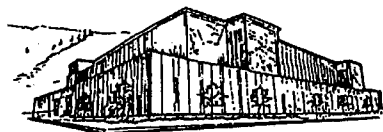
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**ACQUISITION AND EVALUATION OF
SEDIMENTOLOGIC, PALEOMAGNETIC, AND
GEOCHEMICAL TIME-SERIES DATA FROM FLATHEAD
LAKE, MONTANA: IMPLICATIONS FOR LATE
PLEISTOCENE AND HOLOCENE PALEOCLIMATE**

by

Michael Sperazza

B. S., University of Colorado, Boulder, Colorado 1980

M. A., University of Montana, Missoula, Montana 2000

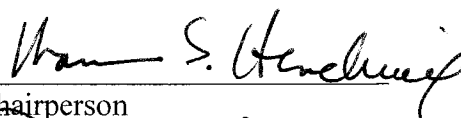
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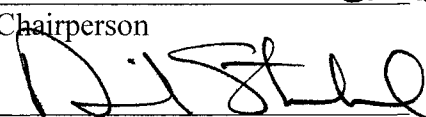
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Acquisition and Evaluation of Sedimentologic, Paleomagnetic, and Geochemical Time-Series Data from Flathead Lake, Montana: Implications for Late Pleistocene and Holocene Paleoclimate.

Chairperson: Dr. Marc S. Hendrix *MSH*

The main objective of this research was to examine a suite of time-series proxy data for potential use in the reconstruction of post Last Glacial Maximum climate history for the northern US Rocky Mountain region. I sought to test the hypothesis that data derived from sediments within a very large watershed ($>18,000 \text{ km}^2$) could provide centennial scale resolution when applied to a basin with multiple climate environs. In this study, I utilized naturally occurring lacustrine sediments from Flathead Lake, Montana to evaluate paleoclimate implications, using a variety of physical, mineralogical, and geochemical data with calculated uncertainty. The results of this research include: 1) development and testing of a methodology for utilizing laser diffraction to determine size fractions of very fine-grained naturally occurring sediments; 2) quantification of methodological uncertainty for paleomagnetic secular variation when used as a chronostratigraphic tool in lacustrine settings; 3) establishment of methodological uncertainty for grain size, mineralogical, and certain geochemical time-series data sets; and 4) evaluation of grain size, mineralogical, carbon/nitrogen, and various elemental analyses and their respective uncertainties for use in paleoclimate reconstructions.

PREFACE

FORWARD

Lake sediment records have long been exploited as a source of time-series data for elucidating past climate changes, because lakes respond quickly to environmental change and may be characterized by long periods of uninterrupted sedimentation. Interpreting ancient climates from lacustrine sediments is achieved by the development of time-series data sets for variable parameters, called proxies, within natural records. A proxy data set is utilized as a substitute for one or more climatic, environmental, or physical condition that existed in the past, but cannot be measured directly. Proxies are presumed to vary predictably with changing climate and so commonly are inferred to represent a record of climate change. Accurate calibration and discernment of these data is essential before they can be used to understand and predict modern and ancient climate variability.

In this dissertation, I collected time-series data from sediment cores recovered in Flathead Lake, Montana. My focus was to quantify the limitations and resolution of the data and to examine proxy variations and uncertainty that can potentially be utilized in paleoclimate reconstructions.

Chapter 1, entitled “High-Resolution Particle Size Analysis of Naturally Occurring Very Fine-Grained Sediment through Laser Diffraction” has been published in the Journal of Sedimentary Research. This paper covers the establishment and testing of operational methods for determining grain size using laser diffraction techniques. The chapter

quantifies uncertainty and the variability of sampling preparation, operational settings and optical properties.

Chapter 2, entitled “Assessment of Paleomagnetic Secular Variation Correlation and Establishment of a Chronostratigraphic Model for Late Pleistocene and Holocene Lacustrine Sediments of Core 9P from Flathead Lake, Montana” evaluates chronological methods applied to core FL-00-9P. A detailed assessment of paleomagnetic secular variation is presented that constrains procedural variations and quantifies uncertainty. A shortened version of this chapter is in preparation for publication.

Chapter 3, entitled “Examination of Potential Paleoclimate Proxies for Large Open Lacustrine Systems: Late Pleistocene and Holocene Sedimentary Record, Flathead Lake, Montana” is an analyses of grain size, mineralogy, and carbon / nitrogen as paleoclimate proxies. The chapter tests proxy resolution and reviews the connection of each to climate change. Potential limitations of each proxy are considered, including uncertainty and external forcing, such as topography, watershed, and sediment redistribution. The chapter concludes with a brief interpretation of climate change, considering uncertainty, for the proxies acquired from Flathead Lake. Various portions of this chapter will be published, including an expanded analysis comparing proxies from other high latitude oligotrophic lakes.

ACKNOWLEDGEMENTS

A project so broad scoped, such as this research, would not be successful without the assistance of a number of individuals and institutions that were instrumental in assisting me with my data collection and the completion of this project. I would like to start by thanking Dr. Marc Hendrix my Committee Chair for his inspiration, motivation, and patient review of my data and articles. I have a special appreciation to Committee member Dr. Johnnie Moore for his challenging perspectives, knowledgeable comments, and laboratory assistance. I give my thanks and gratitude to Dr. Joel Harper, Dr. Jim Sears, and Dr. Randell Skelton, the rest of my Committee, for their reviews of my dissertation and serving on my committee, and Dr. Gray Thompson and Dr. Thomas Foor for their reviews of my documents and comprehensive exams.

The data analyses could not be preformed without material to process and I greatly appreciate the assistance of the University of Minnesota, Limnological Research Center (LRC) for the coring barge and equipment, and for the use of their state of the art facilities to initially process and photograph the recovered cores. I would like to give a personal thanks to Dr. Emi Ito for allowing my use of the facility and Dr. Doug Schnurrenberger, Amy Myrbo, Chad Wittkop, Anders Noren, and Paul Wagner (now University of Montana) for their help and guidance in the lab. And, lastly I would like to thank Dr. Mark Shapley who assisted in all aspects of the coring and become an invaluable friend and colleague. Others that I would like to thank for assistance in coring

include Dr. William Woessner (Univ. of Montana), Dr. Derek Sjostrom (now State of Alaska, Division of Environmental Health), and Melanie Kay.

Processing the core materials and running the laboratory analyses for this much data was an enormous task, for which I have many to thank. I would like to start with Dr. Heiko Langner for the coordination and assistance he provided for myself and many others working on this research when using the equipment in the Murdock Environmental Geochemistry Laboratory in the University of Montana, Department of Geology. Many of those who assisted me with laboratory analyses became good friends and some have expanded their involvement in the project by using some of the Flathead Lake data in their thesis or other research project. I would like to thank those who assisted with the data collection and were inspired to research individual projects, including Shishona Thurston, Thomas Gerber, Sheetal Patel, Donovan Powers, Rachel McCool, Jessie Smith, and Rhuzalene Ortiz-Monclova. I also thank those who assisted with laboratory analyses including Danielle Hughes, Jessie Meyers, Layaka Mann, Nate Stevens, Bryan Nelson, Ben Swanson, Jennifer Butler, Noel Philips, Amy Bondurant, Matt Hertz, Colleen Fitzpatrick, Matt Young, and Matt Affolter. I would also like to acknowledge the generosity of Chevron Corporation for the use of their XRD laboratory in Houston and a personal thanks to Dr. Douglas McCarty for his technical assistance, advice, and hospitality during my visit.

No dissertation project can be completed without the tireless support of Christine Foster and Loreene Skeel in the Geology Office, I sincerely thank them for their help and

support. I also thank Brian Collins for his assistance with computers, GIS, and for being a friend and sounding board during many frustrating moments. I appreciate the encouragement and interest in my research by the rest of the Geology Department's faculty, particularly Dr. Steve Sheriff for his support and assistance with geophysical questions.

This research required skills and expertise in many disciplines and I would like to recognize the following collaborators with whom I have worked and continue to work with on this project: Dr. Cathy Whitlock (Montana State University) and Mitch Powers (University of Oregon) for their work and advise on pollen analyses; Dr. Ken Verosub and Dr. Gray Acton (University of California-Davis) for their work and assistance with paleomagnetic dating; Dr. Sheri Fritz and Dr. Jeffery Stone (University of Nebraska-Lincoln) for their analysis and counsel on the diatom record; Dr. Richard Forester (USGS-Denver) for his work on Ostracodes and Dr. Joseph Rosenbaum (USGS-Denver) for his technical assistance and advice; Ron Hatfield and Darden Hood (Beta Analytic, Inc.) for their work and assistance with problems related to radiocarbon dating; Dr. Chris Fuller (USGS-Menlo Park) for his work on isotopic dating of sediments; Terry Stauffer, Martin Weiland, Dr. Paul Kippax, and Paul Dawson (Malvern Instruments) for their technical and logistical assistance on grain size analyses; Dr. Larry Benson (USGS-Boulder), Dr. Rob Negrini (Cal State-Bakersfield), and Dr. Steve Lund (University of Southern California) who unselfishly provided me with their Secular Variation data; Dr. George R. Rossman (California Institute of Technology) for assistance with mineral

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Financial support for my Doctoral degree and research was critical to my success. I would like to recognize and thank those that provide assistance in this area: The University of Montana Doctoral Teaching Assistantship, National Science Foundation EPSCoR, National Science Foundation grants # ATM-0214273 and # DUE-9950637, SEPM (Donald L. Smith Student Research Grant), Murdock Environmental Geochemistry Laboratory, and Chevron Corporation.

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This study is dedicated to the memory of Nate Stevens and Bryan Nelson (2004), and to my brother B. Paul Sperazza (1992).

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CHAPTER 1

High-Resolution Particle Size Analysis of Naturally Occurring Very Fine-Grained Sediment Through Laser Diffraction

Michael Sperazza, Johnnie N. Moore, and Marc S. Hendrix
Department of Geology, University of Montana, Missoula, MT 59812, U.S.A.
Corresponding author: Sperazza: sperazza@mso.umt.edu

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Abstract

In this paper, we present results from a large number of experiments aimed at quantifying method and instrument uncertainty associated with laser diffraction analysis. We analyzed the size distribution of fine-grained sediment ($< 1\text{-}50\text{ }\mu\text{m}$) from Flathead Lake, Montana, along with samples from local fluvial, volcanic, and soil systems on a Malvern Mastersizer 2000 laser diffractometer. Our results indicate: (1) Optimal dispersion of fine-grained sediment was achieved by adding 5.5 g/l sodium hexametaphosphate for > 24 hours prior to analysis and using 60 seconds of ultrasonication during analysis. (2) Obscuration – a measure of the concentration of the suspension during analysis – produced the most reproducible results at about 20%. (3) Variations in refractive-index settings can significantly alter estimated grain-size distributions. (4) Assumed values for absorption (the degree to which sediment grains absorb the light) can have a profound effect on grain-size results. Absorption settings near 0 resulted in unexpected bimodal grain size distributions for sediments in the $< 10\text{ }\mu\text{m}$ size fraction and significantly skewed the fine-grained tail of coarser samples, probably because of sub-optimal diffraction by particles with a diameter similar in size to the laser wavelength. Absorption settings closer to 1 produced very reproducible results and unimodal grain-size distributions over a wide range of refractive indexes.

Our study has shown that laser diffraction can measure very fine-grained sediments ($< 10\text{ }\mu\text{m}$) quickly, with high precision ($\sim 5\%$ at 2 standard deviations), and without the need for extensive mineralogical determinations. These results make possible a new

generation of studies in which high-resolution time-series data sets of sediment grain size can be used to infer subtle changes in paleohydrology.

Introduction

Grain Size as an Environmental Proxy:

Grain-size distributions of naturally occurring sediment have long been used as an important source of information for the interpretation of sedimentation style (e.g., distinguishing underflow from suspension settle-out deposition) and environmental reconstruction (e.g., distinguishing littoral from offshore settings). Traditional particle size determinations of clay- to sand-size sediment common in offshore lake settings have used a variety of technologies. These include analysis by settling tube, sieve, and pipette (e.g., Beuselinck et al. 1998; Weber et al. 2003), hydrophotometer (e.g., Jordan et al. 1971), electrical sensing (Coulter Counter/Multisizer; e.g., Bianchi et al. 1999), and x-ray absorption (SediGraph; e.g., Singer et al. 1988; Campbell 1998). These technologies can be quite time consuming and relatively imprecise, particularly in the case of sieve and pipette analysis. Reported error for these methods ranges from a few percent in the case of SediGraph technologies (e.g., Campbell 1998) to error in excess of 40% in the case of sieve and pipette methods. Surprisingly, many studies involving grain size analysis fail to report analytic error at all, regardless of the technique, making it difficult to critically assess the value of such data sets.

Over the past few decades advances in laser diffractometry have significantly improved the precision and efficiency of fine-grained particle size analyses. As a result, this

technology is becoming more common as a standard sedimentologic tool (Loizeau et al. 1994; Beuselinck et al. 1998). Unfortunately, however, few published studies involving laser diffractometry analysis of fine-grained, naturally occurring sediments report associated analytical uncertainty. Laser particle diffractometers were originally designed for determination of droplet size in fuel sprays and are commonly used for analysis of synthetic substances such as pharmaceutical compounds and latex paints, all of which have known and consistent optical properties. In contrast, naturally occurring sediments contain diverse mineral compositions, each mineral with a unique set of optical properties that can affect the outcome of laser diffractometry results. Error also can be introduced through variations in sample preparation procedures, the means by which the prepared sample is introduced to the diffractometer, and the machine settings and parameters.

In this paper, we present results from a series of experiments of laser particle size analysis of very fine-grained sediment. Most samples consist of Pleistocene and Holocene sediment from piston and gravity cores recovered from Flathead Lake, northwestern Montana. Flathead Lake is a large (510 km²) open lake that receives > 90% of its sediment from the Flathead River (Moore et al. 1982). Fine-grained sediment is dispersed across the lake bottom through hemipelagic suspension settle-out, with minor redistribution through sediment gravity flows close to the Flathead River delta. Sediment samples we used in this study were collected from cores located well away (~ 19 km) from the delta system, where suspension settle-out sedimentation processes are dominant and median grain sizes generally are < 5 µm. To supplement our experimental analyses of these very fine-grained lacustrine samples, we also included samples of soil, fluvial

sediment, glaciolacustrine sediment, and volcanic ash. Our primary goals in this study were to establish a set of standardized sample preparation procedures and to quantify both method and machine uncertainty associated with laser diffraction analysis of naturally occurring sediment (Table 1).

Table 1–Summary of experimental results conducted in this study.

	<i>Target Test</i>	<i>Tested Range</i>	<i>Analytical Impact</i>	<i>Impact</i>
Sample Preparation	Sonication	0-5 min	Without sonication median grain size is overestimated; excessive sonication may cause aggregation and/or grain fracturing.	Low
	Sub-sampling	Dry, Pipette, Direct	Sub-sampling method may affect median grain size. Consistent methods improve analytical results.	High
Optical Properties	Index of Refraction	RI 1.43-3.22	Little impact for range of natural sediment minerals, if absorption properly set.	Low
	Absorption	0-1	Main optical property, improper setting results in high variability and bimodal distribution in < 7 μm fraction.	High
Machine Parameters	Pump Speed	1000-3000rpm	Stable between 1800 to 2300 rpm. High variability < 1400 rpm, gradual changes > 2300 rpm	Low
	Obscuration	2-40%	Low values and coarser grain size return variable results. Results stabilize in a range of 15-25% obscuration.	Medium

As we describe below, careful application of laser diffraction techniques can result in total uncertainty (i.e., method plus machine error at the 95% confidence interval) of 6% or less for very fine-grained sediments. Such precision makes possible a new generation of sedimentologic studies in which subtle time-series changes in grain size may be used to infer the paleohydrologic history of watersheds.

Laser Diffraction:

Laser diffraction was introduced as a method for particle size analysis in the early 1970s. Early laser diffractometers used a small number of detectors (typically 31 or less) and were somewhat cumbersome to use, some diffractometers requiring lens changes for expanded detection range (McCave et al. 1986; Loizeau et al. 1994; Muggler et al. 1997; Corcoran et al. 1998). These early instruments had a narrow size detection range (i.e., 0.5-560 μm) compared to modern instruments (0.02-2000 μm for the Malvern Mastersizer 2000), resulting in under representation of the very fine grain-size fraction. In addition, early diffractometers used a small number of particle-size bins (e.g., 15) for each analysis, resulting in relatively low resolution of grain-size distributions (McCave et al. 1986; de Boer et al. 1987; Singer et al. 1988; Loizeau et al. 1994; Buurman et al. 1997; Beuselinck et al. 1998). Modern laser diffractometers utilize a larger number of detectors and improved mathematical models, increasing the detection range and distribution resolution, and significantly improving the quantification of particles finer than 10 μm (Muggler et al. 1997; Wen et al. 2002).

Low-angle laser light scattering (LALLS, commonly called laser diffraction) systems typically pass a laser beam of known wavelength through a suspension of the material to be analyzed and measure the angular distribution and intensity of the forward-scattered (diffracted) light by the particles in suspension (Fig. 1). A theoretical model, based on diffraction of particles with particular properties and grain-size distribution, is then fitted to the actual diffraction results. The difference between the measured diffraction pattern and the theoretical diffraction pattern is the portion of the measurement unexplained by the model. Minimizing this residual reduces the analytical uncertainty.

Two main diffraction theories are typically used in the prediction of laser particle size results: Fraunhofer theory and Mie theory. Detailed reviews of these two methods for predicting diffraction patterns along with discussions of the principles of laser diffractometry are presented by McCave et al. (1986), de Boer et al. (1987),

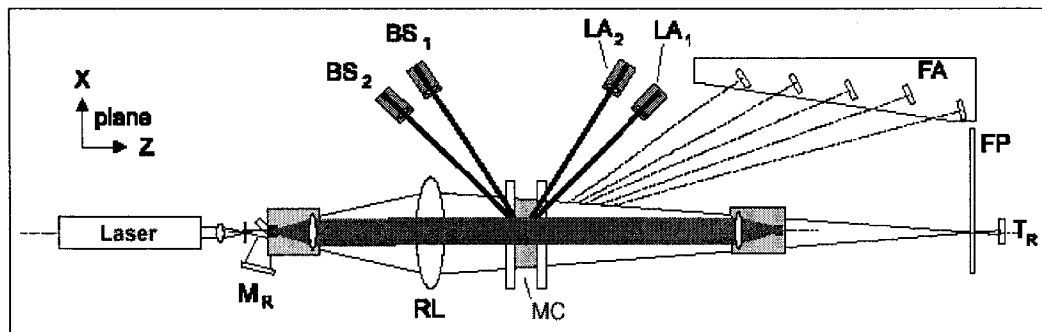


Figure 1—Schematic diagram of Malvern Mastersizer 2000 laser diffractometer. Laser light at a wavelength of $0.632 \mu\text{m}$ is focused by Reverse Fourier Optics (RL) and collected by backscatter (BS), forward angle (FA), and large angle (LA) detectors. Other labeled components are the focal plane detector (FP), obscuration detector (T_R), laser power monitor (M_R), and measurement cell (MC). (Courtesy of Malvern Instruments, Ltd.).

Singer et al. (1988), Agrawal et al. (1991), Loizeau et al. (1994), and Wen et al.(2002) and will not be reviewed in detail here. The basic difference between the two light-diffraction theories is that Fraunhofer theory approximates particle size from extinction efficiency (scatter + absorption) and assumes the same values of extinction efficiency for all particles sizes. In contrast, Mie theory provides for variation in extinction efficiency as a function of particle size (Webb 2000). Hence, Mie-based diffractometry requires that the indices of refraction and absorption be known for both the particles being analyzed and the medium being used to suspend the particles.

Prior to the mid-1980s laser diffractometers relied mainly on Fraunhofer theory because they lacked the computing power to execute the more complicated Mie-based calculations in real time (Wedd 2000). A significant deficiency of Fraunhofer-based grain-size determinations is that they tend to underestimate particle sizes close to the wavelength of the laser source light (McCave et al. 1986; Singer et al. 1988; Agrawal et al. 1991; Loizeau et al. 1994). In contrast, Mie-based calculations are less susceptible to grain-size underestimation near the laser wavelength. As a result of these differences between the two processing techniques, The International Organization for Standardization issued a standardized procedure for the determination of fine-grained particle size distributions, which recommends that Fraunhofer-based diffraction be used only when mean particle sizes are $> 50 \mu\text{m}$ and concludes that Mie-based diffractometry is acceptable for all fine grain fractions (ISO-13220-1 1999; Jones 2003).

Experimental Design

Instrument Operation:

This study was conducted utilizing a Malvern Instruments Mastersizer 2000 laser diffractometer with Hydro 2000MU pump accessory. Two light sources are utilized, a red He-Ne laser at 0.632 μm and a blue LED at 0.466 μm . Diffracted light is measured by 52 sensors and accumulated in 100 size fraction bins. Data were compiled with Malvern's Mastersizer 2000 software version 5. The Mastersizer 2000 takes 1000 readings (snaps) per second. Each measurement run was set to run for 12 seconds or 12,000 snaps. Grain-size analyses reported in this paper are the average of three successive laser diffraction runs (total of 36,000 snaps). Prior to accepting an analysis, we visually inspected the output from each of the three runs for consistency. This method provided a rapid assessment of the potential negative effects of machine spikes, introduction of air bubbles to the suspension being analyzed, or other operational problems. The Mastersizer 2000 utilizes Mie theory to convert the scatter of light energy to grain size and reports grain-size distributions as volume percentage for each size bin. In contrast, sieve and/or pipette methods of grain-size analysis report mass percentage for each size class.

Statistical Representation of Distributions:

Laser diffractometers typically present grain-size distributions as a cumulative curve or a histogram. However, as a convenience when comparing size distributions from different samples, a variety of different statistical measures are available to represent grain-size distributions. Important summary statistical parameters include the mean, mode, and

median grain size, along with percentiles, and other weighted mean values (surface and volume weighted means). Uncertainties associated with these measures are not uniform (Fig. 2), but subject to skewing by low volumetric percentages in non-Gaussian grain-size distributions. For example, a positively skewed grain-size distribution (i.e., larger volume percentage of fine grain sizes) will have more uncertainty associated with the ninetieth percentile, D_{90} than the tenth percentile, D_{10} . For this reason, we focused our uncertainty analysis on median-grain-size measurements, D_{50} . Typically, researchers using grain size as a proxy have found it beneficial to examine changes in multiple measurements (i.e., percent clay, silt, and sand) to enhance the understanding of hydrologic processes (e.g., Campbell 1998). In this study the % sand measure is not a very meaningful quantity for these samples because of the very fine nature of the sediments, $D_{50} \sim 3 \mu\text{m}$.

In this paper we use the term uncertainty as the measure of precision. In all cases we report uncertainty as a 95% confidence interval, approximated as 2 standard deviations above the mean of multiple measurements. To quantify the uncertainty for each of the statistical measures tested, we calculated the mean value and standard deviation for a set of 4-7 replicate analyses. We divided two standard deviations by the mean value for the replicates to calculate the 95% confidence interval as a percentage. We then determined the overall uncertainty for the method/procedure by averaging the 95% confidence intervals for each different sediment sample. Input variables, such as optical settings, equipment settings, and subsampling methods, were tested independently of all other variables.

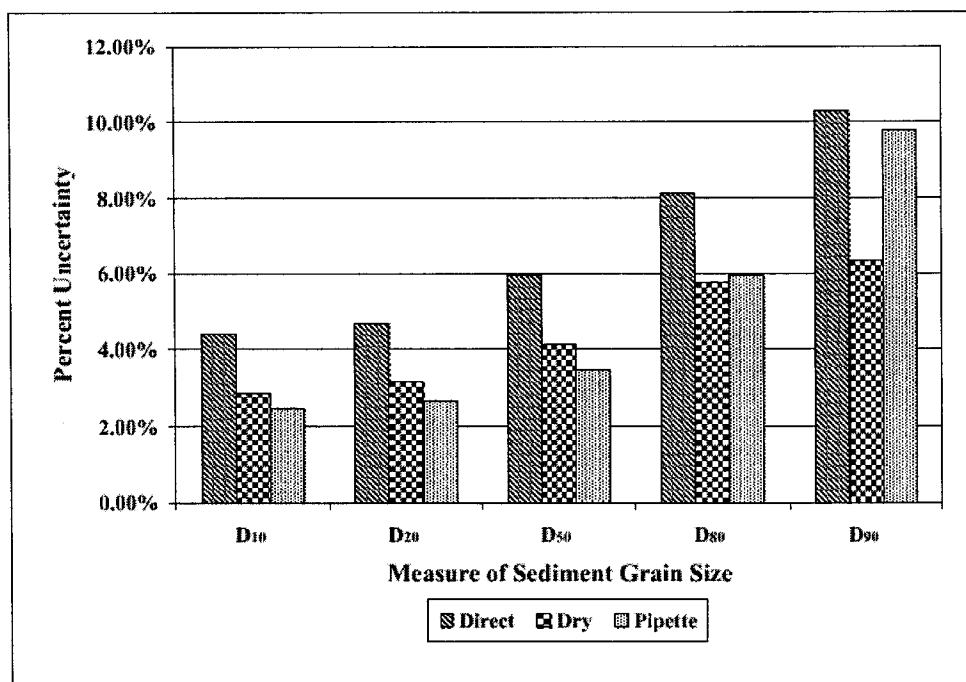


Figure 2—Histogram showing uncertainty as a function of percentile rank of the grain size distribution and aliquot sampling method. Percent uncertainty varies with descriptive measures of estimated grain size, in this case percentile ranks. High uncertainty at high percentile rank, D_{90} , is likely a function of the volume-based measurement of laser diffractometry, where a larger grain represents a larger percent volume than a smaller grain. Unless otherwise noted, in this report we express measured uncertainty for median grain size, D_{50} , at 2 standard deviations. All uncertainties reported are based on data samples listed in Table 2.

Sample Preparation:

Dispersion and Sonication.---The platy clay size fraction has a very high surface-to-volume ratio that increases the net effects of the small interparticle attractive forces. The consequence is that clay has a predisposition to flocculation or agglomeration (McCave et al. 1986). Flocculated clay particles can present a larger target to the optical laser and thus skew the particle distribution towards the larger size fraction (e.g., Chappell 1998). Various techniques have been employed to combat flocculation, including the addition of dispersing agents (Menking et al. 1993; Muggler et al. 1997; Beuselinck et al. 1998) and

variations in the duration of sonication (Loizeau et al. 1994; Chappell 1998). Excessive sonication, however, actually has been reported to flocculate clay particles (M. Weiland, personal communication 2002). We tested dispersion methods that employed sonication. Samples were measured with and without the sonicator that is built into the Mastersizer 2000 instrument. Our objective in applying sonication was to disperse particles while not breaking grains or flocculating the clays. In our experiments, we applied from 0 to 5 minutes of sonication in 10-second increments at two power levels (10 μm and 20 μm tip displacement). Dispersion was assisted with the chemical agent of sodium hexametaphosphate, $(\text{NaPO}_3)_6$. The chemical dispersion also prevented grains from aggregating after sonication and during the grain-size measurements. In all experiments sodium hexametaphosphate was used in a concentration of 5.5 g/l (Tyner 1939; Tchillingrian 1952; Royce 1970).

Subsampling and Aliquot Introduction.--- We use the term "sample" to refer to the bulk sediment collected from soil, outcrop, tephra, or sediment core. In our case, 3" (7.6 cm) diameter lake sediment cores were cut parallel to bedding into slices 1 cm thick. The "subsample" is that portion of the sample that we processed (dried or dispersed in bulk) as part of our aliquot preparation techniques. Each subsample was further divided into 5 to 7 aliquots for replicate analysis. The "aliquot" is the sediment introduced in to the diffractometer.

We explored three different methods of preparing each sediment aliquot and introducing it into the laser diffractometer. Method #1 (hereafter referred to as the "dry" method)

involved drying a sediment subsample in an oven at 50°C for ≥ 24 hours. Aliquots consisting of approximately the same volume of dried subsample were placed in a 30 ml bottle and dispersed for ≥ 24 hours in 5.5 g/l sodium hexametaphosphate. Each aliquot was of sufficient size (~ 0.1 g) to allow the entire bottle to be introduced to the Mastersizer. Method #2 (hereafter referred to as the "direct" method) involved subsampling the sediment core sample with a spatula perpendicular to the depositional laminae. Each core sample was divided into 5-7 aliquots of approximately the same volume and was dispersed in a separate 30 ml bottle with a solution of 5.5 g/l sodium hexametaphosphate for ≥ 24 hours. The contents of the entire aliquot were then introduced directly into the Mastersizer. The direct method is limited to undisturbed sediments where the original laminations are preserved. The third method (hereafter referred to as the "pipette" method) involved placing an entire subsample in a 30 ml bottle containing 20 ml of 5.5 g/l sodium hexametaphosphate. Each subsample for the pipette analysis was volumetrically larger than necessary for an individual measurement in the laser diffractometer. After dispersing for 24 hours, the suspension was vigorously agitated and an ~ 1.25 ml aliquot of the suspension was extracted with a pipette and introduced into the Mastersizer.

To directly compare results among the three aliquot preparation methods, we ran five to seven duplicate measurements per method on the same five samples. The average percent uncertainty for each method was calculated from these results as well as analysis of other samples. Aliquot preparation experiments were conducted only on lacustrine sediments from Flathead Lake.

Optimizing Machine Settings and Initial Experimental Parameters:

In addition to comparing results from the three different methods of subsampling, we conducted a series of experiments on machine settings and initial machine measurements used after the sample is introduced to the diffractometer but prior to the actual grain-size analysis. First, we varied the "density" (measured as the degree of obscuration of the laser beam) of the sample introduced to the Mastersizer, and we varied the speed of the pump that circulates the suspended aliquot within the analysis cell. Second, we experimented with variations in estimated values of refractive index of the sediment and the degree of absorption of the laser by the sediment. These required parameters are used in the Mie Theory calculations for development of the theoretical diffraction pattern that is compared against the actual diffraction pattern.

Obscuration.---The default acceptable range for obscuration on the Mastersizer 2000 is between 10 and 20%. However, Malvern Instruments recommends that with very fine-grained sediments a lower obscuration may be more appropriate (P. Dawson, personal communication 2002). We sought to determine the optimal obscuration range for various types of natural sediments and grains sizes. First, we introduced a high-concentration suspension (40% obscuration) directly into the Mastersizer and measured its obscuration. We then diluted the suspension by adding additional medium (5.5 g/l sodium hexametaphosphate) to reduce the obscuration by ~ 1% before repeating the obscuration measurement. We continued this procedure until obscuration was ~ 2%. Second, we incrementally introduced sediment by pipette directly into the Mastersizer

between measurements; starting with an obscuration of ~ 2% and ending with an obscuration of ~ 40%. For each of these two approaches, we conducted five replicate analyses to examine the effects of obscuration on grain-size results. Samples measured included fluvial, soil, lacustrine, and glaciolacustrine sediments.

Pump Speed.---The Hydro 2000 MU pump unit has variable-speed capabilities to compensate for differences in particle size, density, or sample reservoir volume. In our experiments we utilized a 600 ml beaker with an initial volume of 500 ml of 5.5 g/l sodium hexametaphosphate. We conducted pump-speed experiments by measuring a sediment sample over a range of pump rpm values without removing the sample from the diffractometer. A total of twelve samples were measured once over a pump speed ranging from 1000 rpm and up to 3000 rpm and increased at 100 rpm increments. Samples measured included fluvial, soil, lacustrine, and glaciolacustrine sediments.

Optical Properties:

Index of Refraction.---Determining the primary refractive index (RI) of natural sediments is complicated by the fact that most sediment is a mix of different minerals, many of which have two or more indices of refraction. To estimate the primary refractive index for Flathead Lake sediments analyzed in this study, we performed 82 quantitative x-ray diffraction (QXRD) analyses of core sediment, using the methods established by Środoń et al. (2001). We estimated the primary index of refraction for each sample by summing the product of the refractive indices for each mineral and the percent volume abundance for each mineral as determined by x-ray diffraction. Because some minerals

have two or more indices of refraction, we calculated high, low, and average (weighted and unweighted) indices for each mineral. In addition to using the averaged QXRD data, we performed a series of experiments to further explore the effects of refractive-index extremes on the grain-size distribution. In these experiments, we utilized the refractive index of the mineral with the lowest value (opal from volcanic ash; $RI = 1.43$) and the mineral with the highest refractive index (hematite; $RI = 3.22$).

Absorption.---Along with examining the effects of various input values for RI on grain-size distributions, we studied the effects of varying the value of absorption. Determining the degree of absorption of natural sediments is very difficult, because this parameter changes with particle size and grain shape, chemical alteration of grains, the presence of grain coatings, and the extent of grain surface abrasion. Absorption values range between 0 (perfectly clear grains) and 1 (perfectly opaque grains). In order to examine the effects of absorption on diffractometry results, we held all other sample preparation and machine parameters constant and varied the absorption between 0 and 1. We analyzed the range of absorption values for each of refractive index estimations to determine if absorption had any dependence on RI.

The Malvern Mastersizer 2000 software allows diffraction data to be reprocessed with altered optical settings. In the results presented below, we used software reprocessing to examine the effects of variations in refractive index and absorption on grain-size analysis of lacustrine, soil, fluvial, and tephra samples.

Results and Interpretation

Instrument Precision and Accuracy:

Instrumental precision for the Mastersizer 2000 was very high (~ 1% uncertainty) for samples that were measured 15 times without being removed from the pump unit.

Accuracy of the Malvern Mastersizer 2000 is based on polymer microsphere standard, series 4000, provided by Duke Scientific Corporation. In seven measurements over three years all median grains size results were < 1.2% from the stated standard value of 0.993 μm .

Dispersion and Sonication:

The application of ultrasonic energy as a dispersion agent showed that dispersion increased as additional ultrasonic time was applied to the sample, up to 60 seconds of applied ultrasonic energy (Fig. 3). In some samples, additional ultrasonic energy beyond 60 seconds resulted in a continued slow decrease in grain size, which we speculate may be the onset of grain fracturing by the ultrasonic probe. Some samples showed increases in grain size after 60 seconds of ultrasonic application, interpreted as the flocculation of the clays. From these results, we conclude that the application of 60 seconds of ultrasonic energy with a probe tip displacement of 10 μm , disaggregated samples to achieved maximum stable dispersion for very fine-grained sediments without flocculating the clays or conspicuously breaking grains.

Aliquot Introduction:

We calculated total uncertainty for each of the three aliquot methods based on a total of between 36 and 60 grain-size measurements for each aliquot method (Table 2). Sediment samples for this portion of the study were all from lake cores. Uncertainties of median grain size for aliquot preparation and introduction are 6% or less at 2 standard deviations (dried = 4.1%, direct = 5.9%, pipette = 3.5%; Table 2). We found no consistent statistically significant differences among all samples analyzed by the aliquot sample preparation methods (Fig. 4). However, we did observe several significant trends among some samples. First, three of the five samples prepared using the dry method show a trend of significantly coarser grain size for the D_{10} and D_{50} fractions (Fig. 4A, B). We interpret these results to reflect the formation of particulate aggregation (cakes) created by the drying process. Second, two of the five samples introduced by pipette displayed significantly finer grain sizes in the D_{10} and D_{50} size fractions (Fig. 4A, B).

We have also observed this trend in other analyses of fine grained sediment (Sperazza et al. 2002) and attribute it to heterogeneous suspension during the pipette sampling process and the tendency for the pipette to preferentially sample grains with lower settling velocities. Third, one of the five samples prepared by the direct method display significantly coarser grain sizes in the D_{90} fraction (Fig. 4C). On the basis of these experiments, we conclude that the statistically significant grain size differences associated with some of the dried and pipette samples are in part a function of the procedure itself. Many grain-size studies have used methods that involve either pipette or dry subsampling (e.g., Loizeau et al. 1994; Muggler et al. 1997; Chappell 1998).

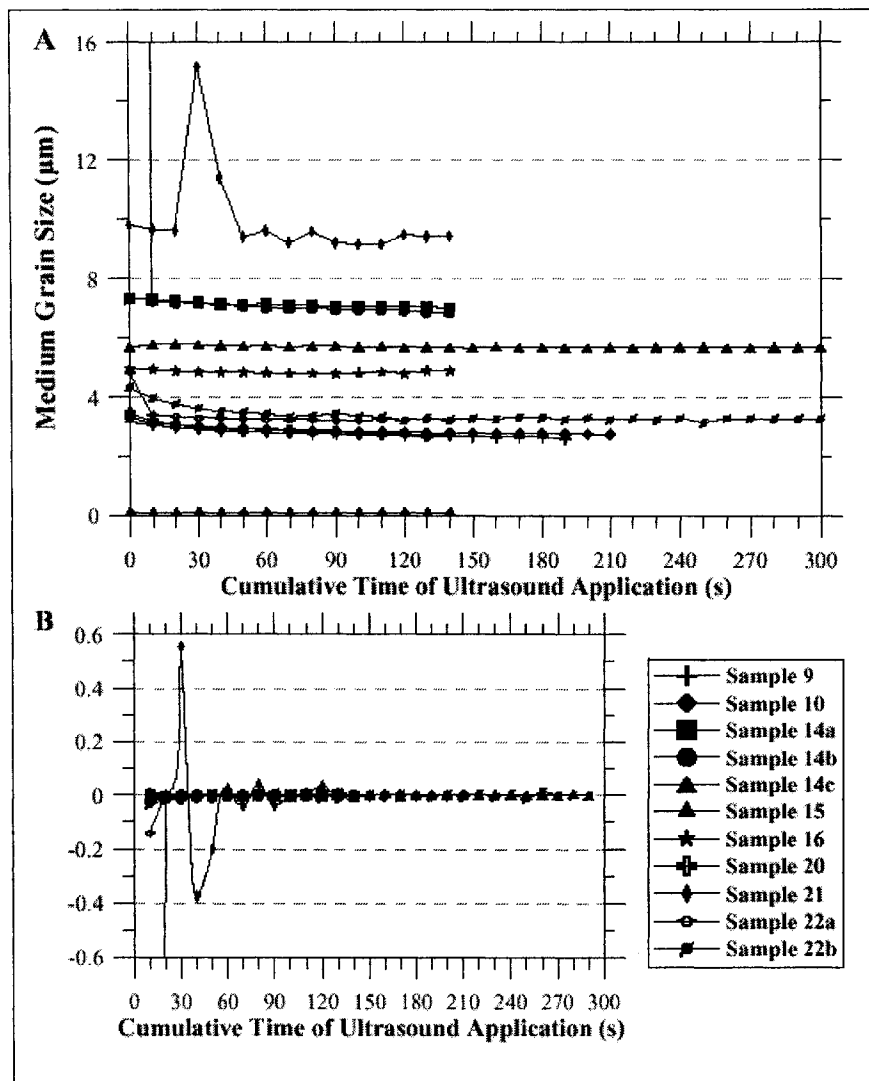


Figure 3— Results on median grain size as a function of ultrasonic energy for dispersion. A) Eight samples were subjected to an increased cumulative period of ultrasonication at 10 second intervals, and two of the samples were replicated. B) First-derivative curves for each analysis show that after about 1 minute of ultrasonication, median grain size appears stable for all samples. Samples did not aggregate with increased duration of ultrasonication. All samples analyzed are lacustrine sediments using 10 μm tip displacement ultrasonication.

Although our data suggests that statistically significant differences can exist among the different preparation methods, the high reproducibility of each individual technique

(<6%) is such that quantitative determinations of time-series change are achievable provided that a consistent sample preparation method is used throughout the study. The higher uncertainty (5.9%) of the direct aliquot method was an unexpected result (Fig. 2). These aliquots were taken from 1-cm-thick slices of undisturbed lake core cut parallel to depositional laminae. The aliquots themselves were cut perpendicular to bedding and

Table 2 –Calculated uncertainty for the three aliquot introduction methods.

Measurement	Direct Method	Dried Method	Pipette Method
D₁₀	4.4%	2.9%	2.5%
D₅₀	5.9%	4.1%	3.5%
D₉₀	10.3%	6.3%	9.8%
% Clay	5.7%	4.0%	3.1%
% Silt	2.5%	1.7%	1.8%
% Sand	73.3%	37.1%	97.5%

n range	5-7	4-7	6-8
Total n	36	47	60
Samples	6	8	9

Uncertainty is calculated as the average percent of two standard deviations. Shown are subsets of the possible descriptive measures that can be used to describe the grain size of a sample. The lower part of the table shows the number of measurements used to calculate the uncertainty.

included the entire thickness of the core slice. We interpret that, despite the hemipelagic nature of the deposits and the large distance from the sediment source, each core slice was characterized by a laterally heterogeneous grain-size distribution that was within the detection range of our experiments. These results suggest that mixing a larger subsample, as required by the dry or pipette methods, may promote homogenization of the subsample and subsequently improve uncertainty at the aliquot level. Alternatively,

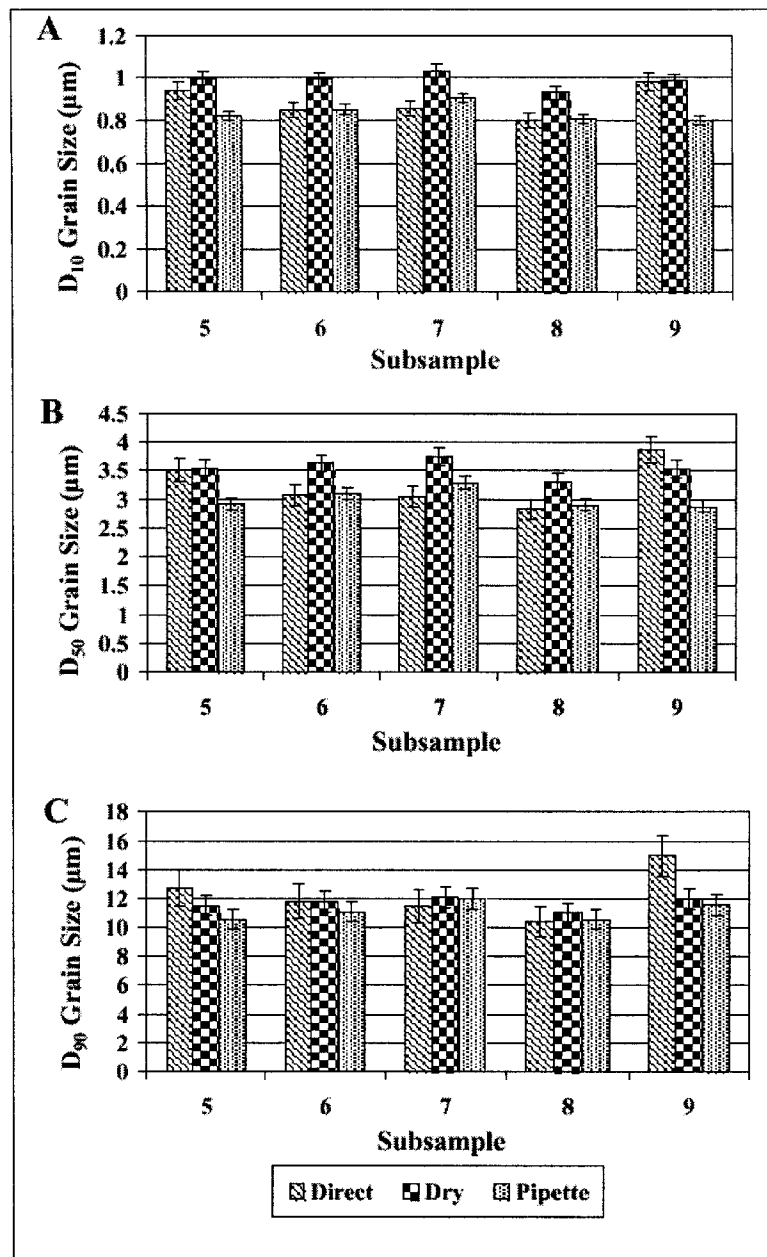


Figure 4—Aliquot subsampling uncertainty for the three subsampling methods. Data for each method is based on the same five samples. Shown are three percentile measures: A) D_{10} , B) D_{50} , and C) D_{90} . Error bars reflect uncertainty at 2 standard deviations for each percentile reported in Table 2. All samples are offshore lacustrine sediments.

improved uncertainty of the dry and pipette methods may be an artifact of incomplete sampling or alteration of the entire grain-size spectrum as discussed above.

Obscuration:

Optimal obscuration occurs when a sufficient number of suspended particles are present to significantly diffract the laser beam but the suspension is not so dense to render the suspension impenetrable by the laser light. We tested the effect of obscuration on median grain size over a range of 2 to 40%. Our obscuration results show that values less than 5% produced poor precision and unpredictable trends (Fig. 5). For very fine-grained sediments (median grain size $< 10\ \mu\text{m}$) the median grain sizes were somewhat higher at lower obscuration values than at higher values (Fig. 5A). With increased obscuration, median grain size values for very fine-grained sediments continue to decrease slowly but are essentially stable between 15 to 20% obscuration (Fig. 5B). Hence, we adopted 15 to 20% range as a working obscuration target for our standard operating procedure. Coarser grained sediments (median grain size $> 10\ \mu\text{m}$) exhibited more erratic behavior, with substantial changes in calculated grain size when obscuration was $< 15\%$ (Fig. 5). Although the curve for coarse-grained sediment flattens in the 15 to 25% range, it has higher variability in that range and at greater obscurations than the very fine-grained sediments. This may be due in part to the use of the additive method of measuring obscuration (see aliquot introduction section above).

Pump Speed:

We examined the effects of variations in pump speed on resulting estimated grain-size distributions. The turbulence created by the Mastersizer pump propeller keeps the

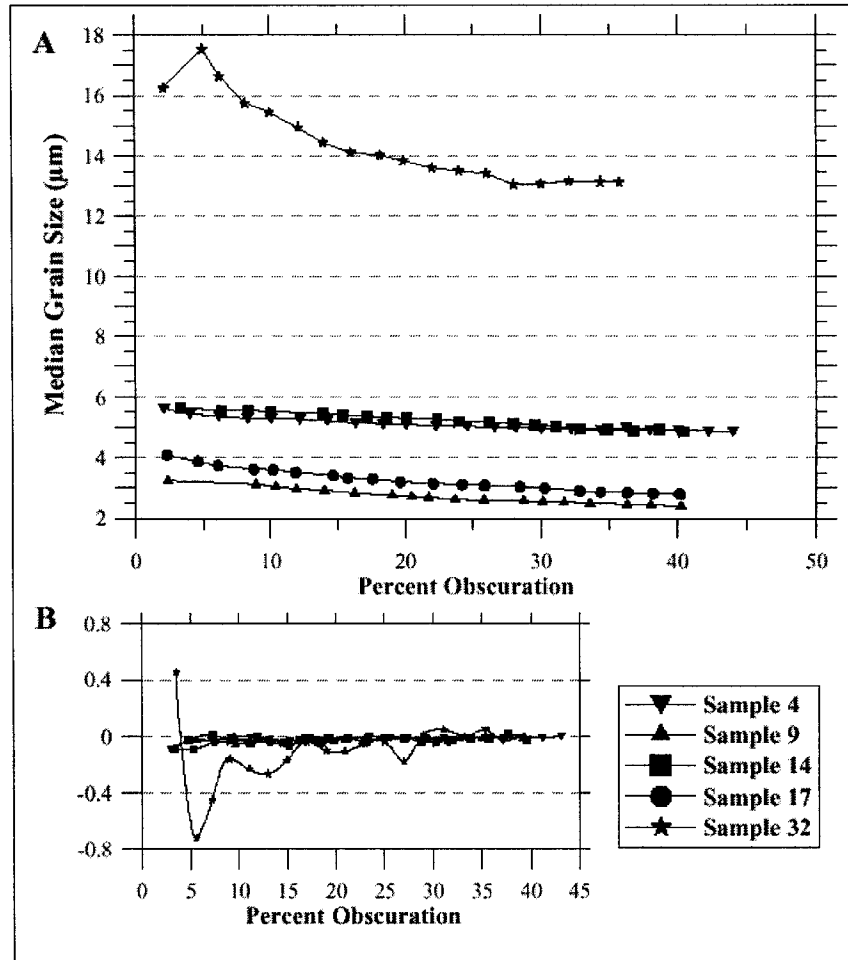


Figure 5—Results of obscuration experiments for very fine-grained and fine-grained sediments. A) Estimated median grain size for all samples decreases with increased obscuration. B) The first-derivative curves show median grain size for finer sediments stabilize with low variability using an obscuration range of 15 to 20%. Results on median grain size from coarser sediments are more variable, likely due to difficulty maintaining a uniform suspension (B). Variability for all samples is highest at obscuration values < 7% (B). Sample 4 is glacial lake sediment, sample 32 is river sediment, and the remaining samples are lacustrine sediments.

sediment sample in suspension. Our analyses show that very fine-grained sediments ($< 10\ \mu\text{m}$) were stable over the entire range examined (1000 to 2500 rpm, Fig. 6). Fine-grained sediment ($\sim 12\ \mu\text{m}$) was stable over the range of 1600 to 2300 rpm, with reduced precision on the tails of the examined range. Coarser samples ($> 40\ \mu\text{m}$) did not stabilize at pump speeds < 1800 rpm. At pump speeds of > 2300 rpm coarse-grained samples ($> 40\ \mu\text{m}$) gradually fined or coarsened slightly (Fig. 6A). We conclude that the turbulence of the pump was not sufficient to maintain the suspension of the coarser sediment at the lower pump speed. Optimal results were achieved with pump speeds between 1800 and 2300 rpm. In our experiments we maintained a pump speed of 2000 rpm.

Index of Refraction and Absorption:

The primary difference between the Mie and Fraunhofer theories is that Mie utilizes the index of refraction (RI) and absorption (ABS) of the sediment and liquid medium.

Unlike the industrial applications of laser diffraction, the optical properties of natural sediments are highly variable and usually unknown to the researcher. As a starting point we used standard methods provided by the instrument manufacturer to determine the proper optical property settings for samples in which the indices of refraction and absorption were unknown. This necessitated reprocessing data using the Mastersizer software and entering different values of refractive index and absorption until the lowest weighted residual value was achieved. This procedure resulted in values for our lacustrine sediments of $\text{RI} = 1.52$ and $\text{ABS} = 0.0$, the values for typical glass beads. Using these optical settings, however, our results showed strongly bimodal grain-size

distributions, which we viewed as unlikely for hemipelagic lacustrine sediments collected 19 km from the sediment source.

To independently develop a set of estimated RI values, we obtained data on mineral composition by QXRD for 82 lacustrine sediment samples collected over a 7 m section of core from Flathead Lake. Using the average, minimum, and maximum percentages for each mineral identified in the QXRD analysis and the minimum and maximum RI values for each mineral, we calculated a set of weighted-average refractive indices (Table 3).

We calculated the low and high RI averages, with and without the RI outlier value of 3.22 (hematite).

Table 3—Average mineral composition from 82 QXRD measurements of Flathead Lake core samples and the minimum and maximum values of index of refraction for the representative mineral for each group.

<i>Mineral Group</i>	<i>Avg. %</i>	<i>Max %</i>	<i>Min %</i>	<i>RI Mineral</i>	<i>RI Min</i>	<i>RI Max</i>
Quartz	27.5	32.7	18.6	Quartz	1.543	1.554
Kspar	2.8	4.1	0.7	Microcline	1.518	1.525
Plagioclase	5.4	10.2	1.1		1.528	1.542
Calcite	2.7	15.0	0.0	Calcite	1.486	1.660
Mg-Calcite	0.3	1.0	0.0		1.500	1.700
Dolomite	2.5	10.8	0.0	Dolomite	1.500	1.681
Halide	0.1	0.8	0.0	Halite	1.544	1.544
Pyrite	0.2	0.9	0.0	Pyrite	1.810	2.000
Siderite	0.2	0.9	0.0	Siderite	1.570	1.788
Opal	0.1	5.5	0.0	Opal	1.430	1.430
Fe (oxy-)hydroxide	0.6	1.2	0.0	Hematite	2.940	3.220
Kaolin	1.0	4.5	0.0		1.715	1.728
2:1 Al Clay	47.6	70.7	21.8	Illite	1.530	1.600
Fe-Chlorite	2.2	9.5	0.0	Chinocllore	1.571	1.599
Mg-Chlorite	7.1	12.8	0.0	Dozyite	1.570	1.580

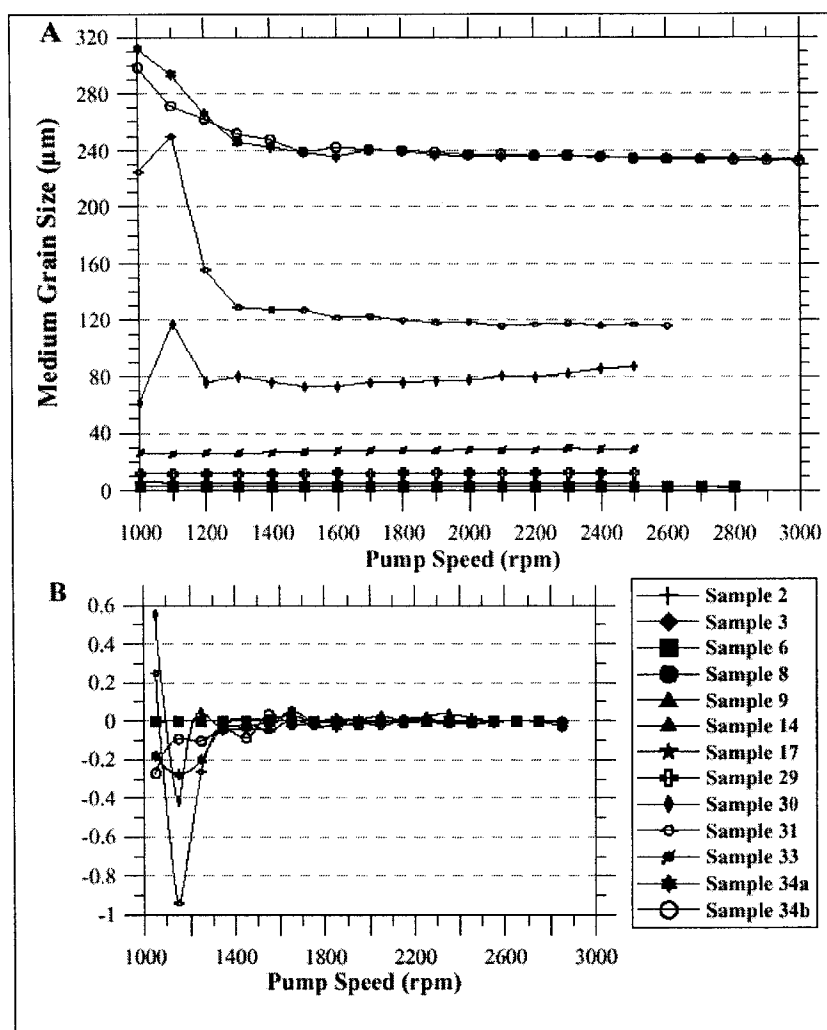


Figure 6—A) Results of estimates of median grain size as a function of pump speed. **B)** First-derivative curves show that fine-grained sediments yield stable results for grain size over a broad range of pump settings whereas coarser grain sediments yield more variable estimates of grain size. Samples 2 and 3 are glacial lake sediments, samples 6 – 17 are lacustrine sediments, samples 29-31 are river sediments, and 33 and 34 are soil sediments.

Additionally, we used the refractive-index extremes of the minerals present in the sediment (opal, 1.43; hematite, 3.22) despite their low volumetric abundance. In this paper we report data on RI values of 1.43, 1.52, 1.57, 1.60, 1.78, and 3.22.

We measured numerous very fine-grained sediment samples with these RI values using an absorption value of 0.0. The overall results are highly variable, ranging from unimodal to bimodal distributions (Fig. 7A). Median grain-size variability was as high as 5.9% with an uncertainty of > 13%. Variability in the finest size fraction, D_{10} , showed even higher variability of ~ 9%. Using these RI and absorption settings, we observed bimodal distributions in numerous samples of very fine-grained lacustrine sediments. Coarser-grained fluvial and soil sediment samples did not display bimodal distributions, but displayed some variability within the < 7 μm fraction (Fig 8). Grain-size variability for coarse samples was < 1% at D_{50} and ~ 2% for the D_{10} fraction.

The internal absorption is a highly variable property between minerals, ranging from totally opaque (e.g., pyrite) to optically transparent (e.g., some quartz). Our software processing had identified an optimal absorption value of 0.0 for the lacustrine sediments, so we used this value in our initial RI experiments. However, we suspected that these sediments did not actually have the same absorption value as clear glass, so we experimented with varying absorption values ranging from 0.0 to 1.0. Higher absorption values decreased scatter in estimated grain size and produced a more unimodal distribution as the absorption setting approached 1 (Fig. 7A-F). In some cases, a substantial reduction in the bimodality of a grain size distribution was achieved by increasing the absorption value from 0.0 to as little as 0.001. These results were consistent for the entire range of tested RI values (1.43-3.22). Figure 7A shows a typical highly variable distribution for a sample over the examined RI range when absorption is

set at 0.0. As absorption values are increased, the distribution becomes more unimodal and stable (Fig. 7B-F).

These results are supported and refined by the calculated weighted residual and sediment concentration. The weighted residual is lowest when the absorption is set at 0.0, but it starts to rise at a value of 0.001, stays high through 0.5, and then declines through a value of 1. The reduction of the weighted residual value as absorption is increased towards a value of 1 support the appropriateness of the high-absorption setting. High-resolution measurements by weight of actual sediment concentrations also support a value of 1 for absorption. As absorption reaches a value of 1, the values for sediment concentration calculated by the software are closest to our measured sediment concentrations.

To independently verify our absorption findings we conducted the same experiments utilizing a BCR Quartz Reference Particles standard (BCR 66) from Duke Scientific Corporation. This standard has a certified distribution and known index of refraction (BCR-information 1980; Konert and Vandenberghe 1997). In these experiments involving the standard, absorption values of < 0.1 yielded strongly bimodal distributions. Absorption values between 0.1 and 0.7 resulted in unimodal distributions but more variable grain-size estimates.

At absorption values > 0.8 , distribution curves converged with very low variability. We conclude that the convergence of the grain-size distribution curves, concentration data, and weighted residual values reflect that as the absorption value approaches 1.0, it

approaches the true value for sediment being examined. At absorption settings ≥ 0.9 for mixed mineral compositions the difference from varying RI values is negligible (Fig. 7E, F).

Estimated grain-size distributions were highly dependent on values of absorption setting for analyses of natural sediment. We found that, even with a known index of refraction, values of absorption close to 0 commonly resulted in unreasonable, strongly bimodal grain-size distributions. In each case, the "low" point in the curve was consistently below $10\text{ }\mu\text{m}$; in many instances, distributions showed an absence of any grain-size data between 0.5 and $0.9\text{ }\mu\text{m}$ (Fig 7A). As the input absorption value approached 1, the resulting grain-size distribution evolved into a much more reasonable unimodal distribution. Interestingly, none of the coarser sediments that we analyzed in this study (fluvial, soil, volcanic ash) produced bimodal distribution for low absorption values (Fig. 8). However, variations in the grain-size distribution did occur in the $< 7\text{ }\mu\text{m}$ size fraction at low absorption values, suggesting that the estimated grain-size distribution for all natural sediment types were affected by variations in entered values of absorption. Bimodality occurs when a significant volumetric percentage of the sample is in the very fine-grained fraction and typically when the median grain size of the sample falls below $10\text{ }\mu\text{m}$. This issue significantly affects the precision of grain-size distribution data sets and seems to be a function of particle size and shape in the range of the laser light wavelength.

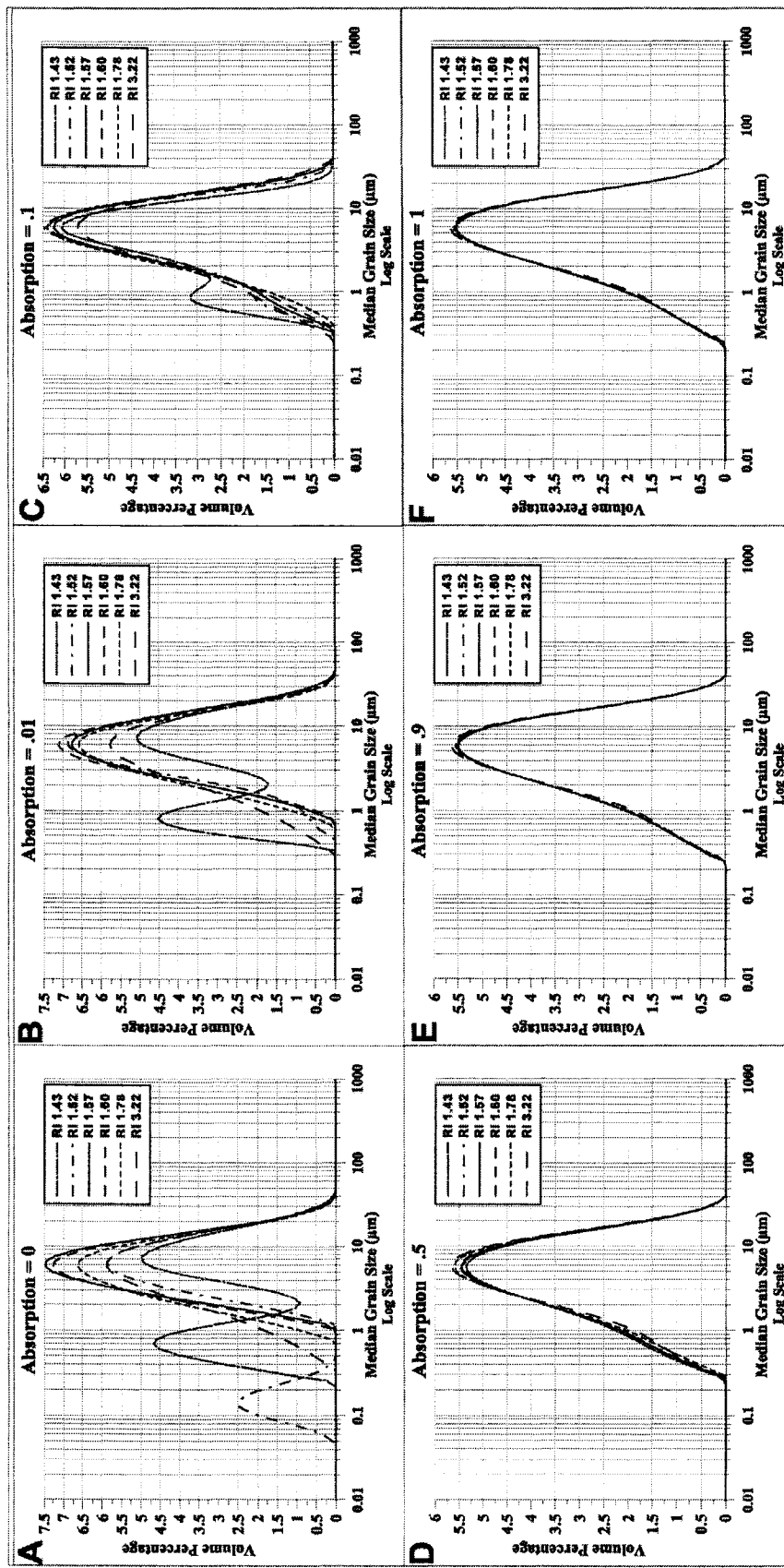


Figure 7—Results on grain-size distribution showing the strong dependence on input value for absorption for Mie-theory-based computation of grain size in a single sample of lacustrine sediment. In these representative series of graphs (A-F), ranges of refractive indices are charted at different absorption value settings. In chart A, bimodal distributions are present and variability is high. As the absorption value is increased (B-F), bimodality and variability both are reduced. When absorption settings reach 0.9 or greater, distribution curves converge and RI settings become less significant.

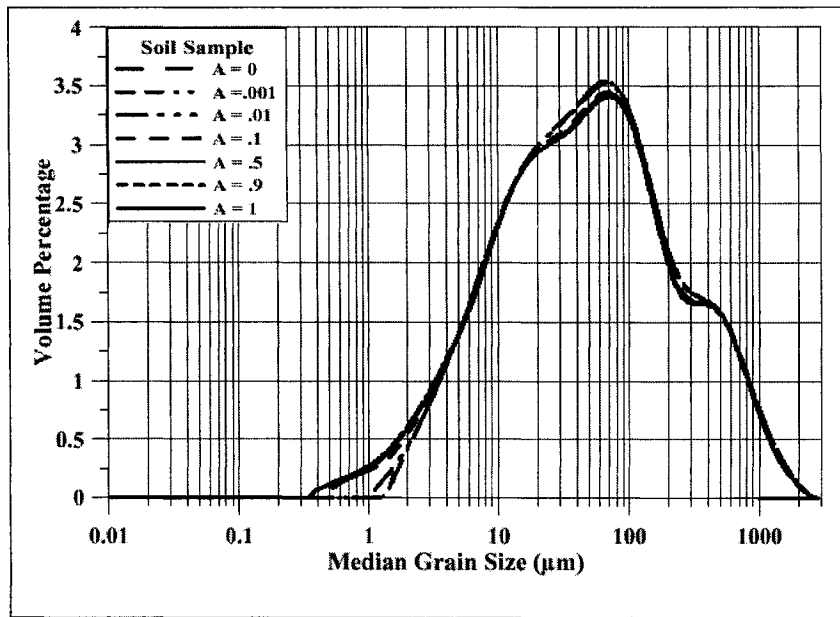


Figure 8—Grain-size distribution showing an example of how variation in input values of absorption can affect the fine tail of coarser-grained samples. Note how the fine tail ($< 7 \mu\text{m}$) of this soil sample is extended with higher input values of absorption. Refractive index for all measurements is 1.57.

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CHAPTER 2

Assessment of Paleomagnetic Secular Variation Correlation and Establishment of a Chronostratigraphic Model for Late Pleistocene and Holocene Lacustrine Sediments of Core FL-00-9P from Flathead Lake, Montana

Abstract:

The establishment of chronostratigraphic models in lacustrine settings often relies heavily upon the acquisition of radiometric carbon dates. Unfortunately, however, this dating method is limited in some lacustrine environments, particularly oligotrophic lakes with poorly preserved organic material. An alternative dating method that has been gaining acceptance in lacustrine, marine and terrestrial research is paleomagnetic secular variation. The two components of secular variation, inclination and declination, provide a time-series record of magnetic pole wandering that has been correlated globally (e.g., Noel and Batt 1990; Butler 1992). Secular variation records from well-dated localities can be used to establish a chronostratigraphic framework for inclination and declination variations. In this study we sought to examine the limitations of secular variation as a dating method and quantify its methodological uncertainty as applied to piston cores recovered from Flathead Lake, Montana. We found that interbasinal and extrabasinal secular variation records can be correlated with an uncertainty of $\pm 6.2\%$ and $\pm 10.4\%$, respectively. The total uncertainty for the method is a function of the reference record data set quality and methods used to interpolate the timing of tie points in the paleomagnetic record that fall between directly dated stratigraphic horizons. The compounded effects of these methodological uncertainties limit resolution of the secular variation chronologic technique to approximately 300-500 years for Flathead Lake cores.

Introduction:

In this study, I describe the techniques and procedures used to establish a chronostratigraphic model for fine-grained, organic-poor sediment from piston core FL-

00-9P (9P), recovered from Flathead Lake, Montana. Flathead Lake is a large oligotrophic lake located at the southern end of the Rocky Mountain Trench in northwestern Montana. Flathead Lake sediments have proven difficult to date partly because of low recovery of large ($> 125 \mu\text{m}$) organic fragments (none have been recovered from core 9P) and a significant reservoir effect from dead carbon that significantly reduces the precision of bulk carbon dating approaches. The maximum total carbon (TC) in the sediments is $< 4\%$ wt and typically $< 2\%$ wt, with total organic carbon (TOC) rarely exceeding 1% wt. (Sperazza et al. 2003).

Piston core 9P is 7.10 m in length and was recovered from a position in the central part of Flathead Lake, 19 km from the mouth of the Flathead River (Fig. 9B). The lowermost 0.90 m of the core consists of a series of centimeter-scale upward fining successions (see Fig. 9C) with very low organic carbon values (average $\sim 0.2\%$ TOC) and very fine grain size (median $\sim 1.6 \mu\text{m}$). These centimeter-thick upward fining sequences pass upsection into 6.2 m of weakly laminated silt with a median grain size of $\sim 4 \mu\text{m}$ and slightly higher organic values (average $\sim 0.4\%$ TOC). Core 9P contains two tephra deposits, which were sent to the Geoanalytical Laboratory at Washington State University for geochemical profiling (Appendix A). Analysis of major and minor element distributions in tephra glass confirmed, with a 99% confidence level, that these were the Mount Mazama Crater Lake tephra (340-375 cm) and the Glacier Peak G tephra (610-611 cm). Calibrated ^{14}C dates of $7,630 \pm 80$ cal. yr. BP have been established for Mt. Mazama (Zdanowicz et al. 1999) and $13,180 \pm 120$ cal. yr. BP for Glacier Peak (Hallett et al. 2001) by acquiring

multiple ^{14}C dates in organic horizons above and below deposits of these two volcanic ashes as they occur in various localities around the Pacific Northwest.

By far the most common dating method for late Pleistocene and Holocene sediments is radiometric ^{14}C dating (Wagner et al. 2000; Rosenmeier et al. 2002). However, in many natural settings the abundance of datable organic material is significantly limited or simply is nonexistent. In these instances, some researches have utilized radiometric dating of bulk sediment (Colman et al. 1996; Rashid et al. 2003), pollen (Brown et al. 1989), and carbonates (Dean and Megard 1993; Makhnach et al. 2004; Yi et al. 2004) as source material for radiometric ^{14}C dating. However, these sources of datable material are prone to reservoir effects, which can increase the reported ^{14}C age (Doran et al. 1999; Geyh et al. 1999; Dumond and Griffin 2002).

Recent advances in dating have been made for sediments in which carbon dating has been problematic or inconclusive. Some of these alternative techniques include varve counting (Anderson et al. 1993; Brauer et al. 1999), thermal luminescence (Shulmeister et al. 2001; Spencer and Owen 2004), uranium series dating (Szabo et al. 1995), electron spin resonance (Molodkov 1993), cosmogenic exposure dating (Jackson et al. 1999; Briner et al. 2003), and correlation of paleomagnetic secular variation time-series data sets (Verosub et al. 1986; Hagee and Olson 1989; Zic et al. 2002).

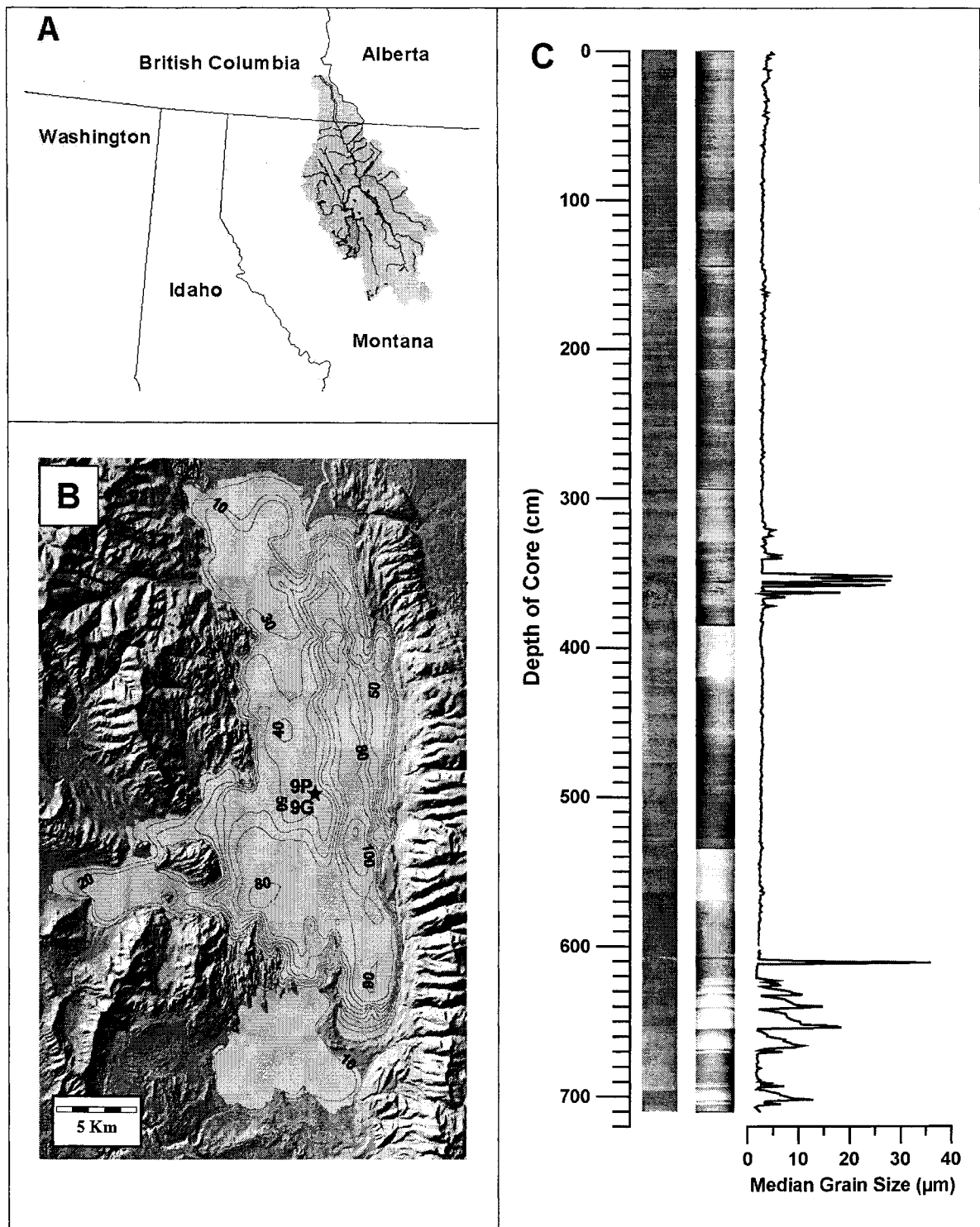


Figure 9 – A) Location map showing the Flathead Lake (Blue) watershed in northwestern Montana and adjacent parts of Idaho and British Columbia, Canada. B) Map of Flathead Lake showing location of cores 9P and 9G. Bathymetry in meters. C) Core 9P composite photo and X-radiograph with median grain size data. Note the presence of tephra at 345-375 cm and 610 cm, and the upward-fining sequences below 620cm.

Initial Chronologic Controls:

Top of Core:

Piston cores commonly disturb the uppermost water saturated sediments, and some analysis is required to determine whether any portion the top of the core is missing. In the case of core 9P, the well-developed oxidized layer seen in gravity cores from Flathead Lake was not recovered, suggesting that the youngest portion of the sediment record is missing. The youngest recovered sediments in core 9P and core FL-00-9G (9G), a companion gravity core recovered simultaneously with 9P and located approximately 1 m away from the 9P drive site, are hemipelagic and likely far from any bottom hugging traction transport-influenced flow more common near the Flathead River mouth. The grain size records of these cores thus are expected to exhibit similar trends that could be correlated to estimate the thickness and duration of time represented by unrecovered sediment at the top of core 9P. Importantly, core 9G has been dated by ^{137}Cs and ^{210}Pb isotope methods by Chris Fuller at the US Geological Survey in Menlo Park, CA (Fig. 10A & B), thereby providing a quantitative basis for estimating the length of time missing from the core 9P sediment record. To correlate the grain size between the two cores, I shifted the 9P curve until the correlation coefficient between both data sets was maximized (Fig. 10C & D). In general, correlation coefficients between the two cores are low (max. $R^2 = 0.354$) likely due to differences in sampling methods (direct method for 9P and dry method for 9G, see Sperazza et al. 2004) and compressional stresses on the gravity core (9G). Whereas the piston cores are transported, processed,

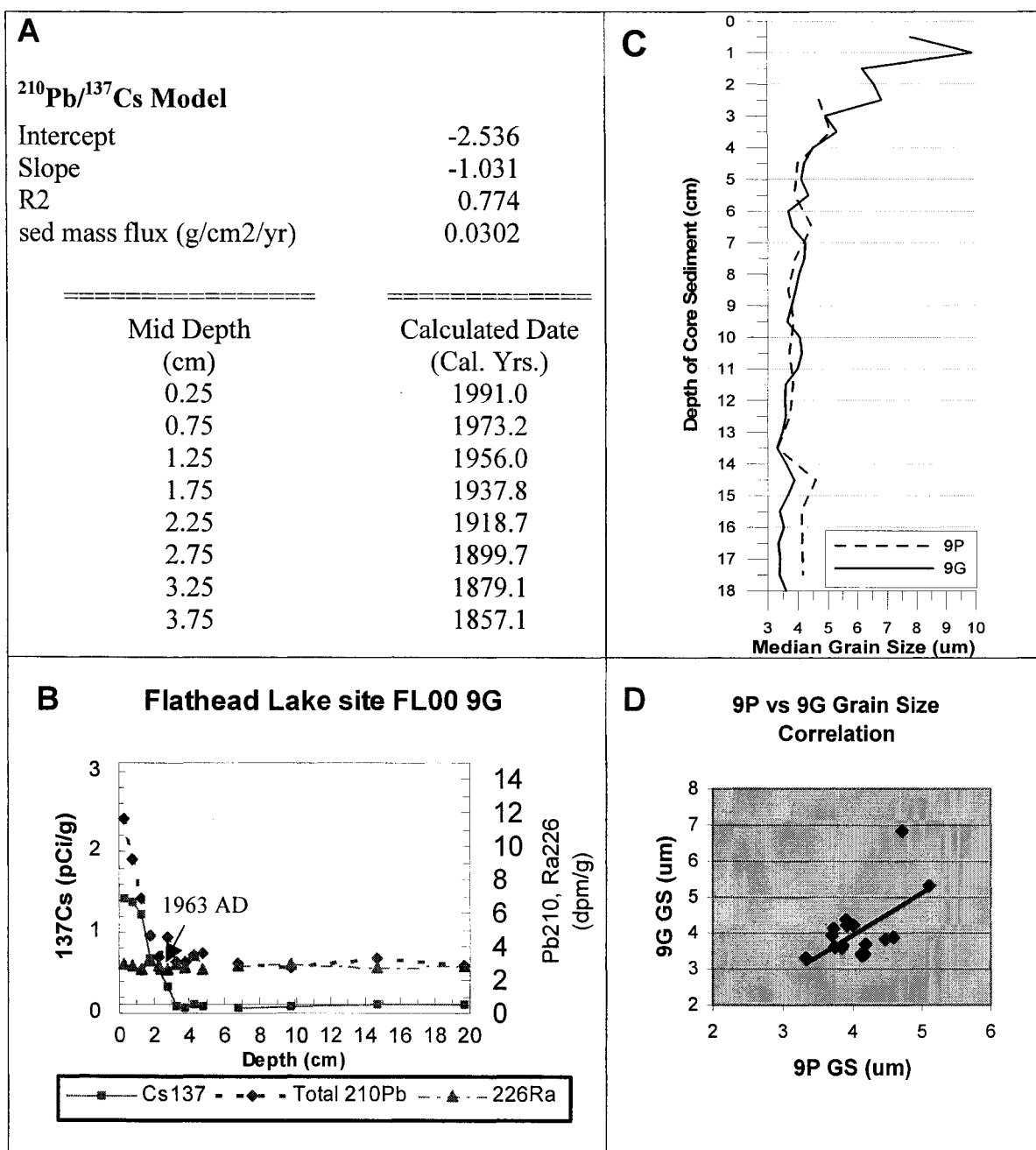


Figure 10 – Dating model for core 9G based on ^{137}Cs and ^{210}Pb methods (A). Date calculated is for the midpoint of the 0.5 cm intervals. Dates were then extrapolated from mid-depth to bottom of interval. (B) Graph plotting isotopic data vs. depth distribution of ^{137}Cs and ^{210}Pb for core 9G (^{226}Ra used as a baseline for ^{210}Pb data). Data in panels A & B was provided by Chris Fuller (USGS). (C) Determination of the top of core 9P is based on correlation of median grain size to core 9G. Statistical best fit of grain size suggests top 2.5 cm of core 9P is missing due to over penetration (C). Correlation coefficient is low ($R^2=0.35$) due to different sampling methods and compressive forces on the cores.

and stored horizontally, the gravity cores are transported and stored vertically, making them susceptible to dewatering and compaction. Additionally, gravity cores are extruded and sliced at half-centimeter intervals, which can apply an undetermined amount of compressional stress to the sediments. From the grain size correlation data, I estimate that the top of core 9P was roughly 2.5 cm below water/surface interface. Accordingly, the effective date for the bottom of the first one centimeter interval of core 9P (FL-00-9P-I-1) is 1908 AD or 92 cal. yr. BP, based on the isotope dating of core 9G.

Middle of Core

The central part of the core is chronologically constrained by the two volcanic tephtras. The deposit of the younger tephra (Mt. Mazama) is disturbed; it contains four separated deposits of the ash between 340 and 375 cm (Fig. 9C). Elsewhere within Flathead Lake, the Mazama tephra consists of a single layer that is about 12 cm thick and fines upward. The lowermost and uppermost of the four individual Mazama tephra layers in core 9P are characterized by a major and minor element distribution that is statistically indistinguishable from that of the Crater Lake eruption of Mt. Mazama (Appendix A). Between each of the tephra deposits are 1-5 cm thick deposits of apparently laminated hemipelagic sediment (Fig. 11). However, X-radiographs show that the tephtras are internally disrupted and that sedimentary lamina in hemipelagic sediment between tephra layers are not as well preserved as those above and below the zone in which the Mazama tephra occurs. As a result, we cannot determine with certainty whether the hemipelagic deposits represent significant time between tephra layers. The X-radiographs suggest that the hemipelagic sediments between the tephtras are likely disrupted.

Based on this physical sedimentologic analysis, I infer that the entire stratigraphic interval that includes the Mazama tephra layers in core 9P is disturbed. Likely, this disturbance resulted from local slope destabilization caused by the rapid deposition and/or redistribution of the tephra during or immediately after the eruption. Seismic reflection data from the basin do not suggest any large scale redistribution of sediment at this time (Hofmann et al. 2006), suggesting that sediment destabilization was localized to the core 9P site and not a lake-wide phenomenon. Because the Mt. Mazama tephra represents a single time horizon ($7,630 \pm 80$ cal. yr. BP) elsewhere across the Pacific Northwest and our interpretation of X-radiographs from core9P suggest the presence of syn/post-depositional disturbance, we have elected to assign the Mazama date to the entire interval between 340 and 375 cm in this core.

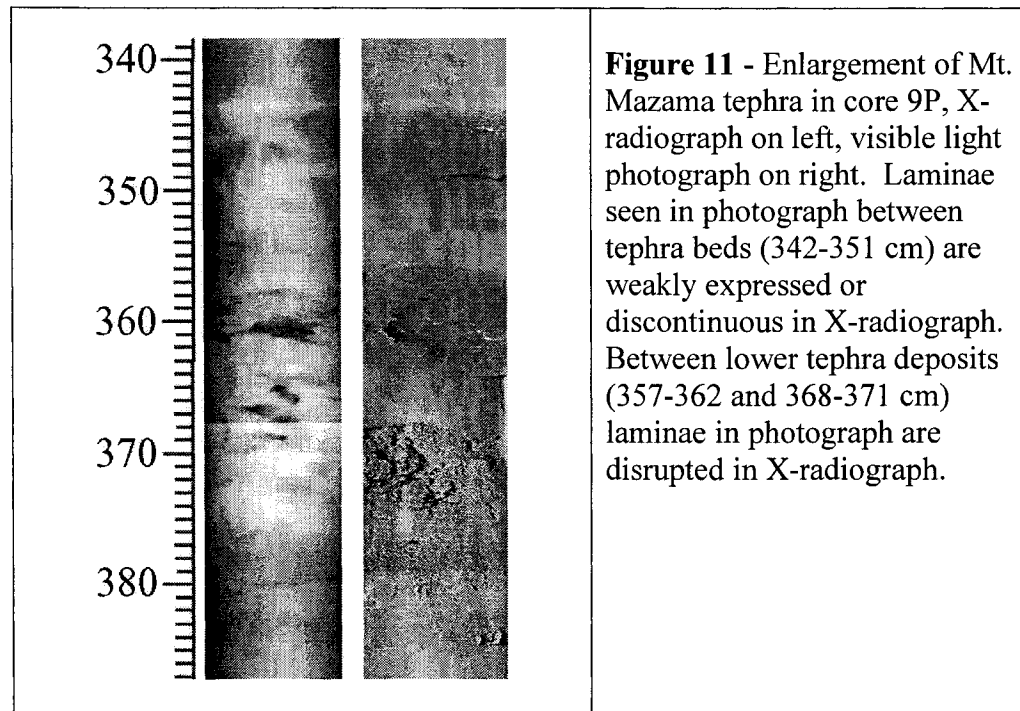
The older tephra occurs from 610 to 611 cm in core 9P and is characterized by major and minor element profiles matching the G tephra from Glacier Peak (Appendix A).

Radiometric dating of organic rich sediments above and below the tephra elsewhere have established an eruption date of $13,180 \pm 120$ cal. yr. BP (Hallett et al. 2001).

Bottom of the Core

In developing the chronostratigraphic model for core 9P I sought to assign a date to the base of each 1 cm core interval. However, no chronologic control was available below the Glacier Peak tephra. Sedimentologically, the lower portion of core 9P contains very-fine grained hemipelagic sediments (~ 1.5 to $2.5 \mu\text{m}$) between 611-621 cm and 672-688 cm. These sediments are similar in grain size and structure to those directly above the

Glacier Peak tephra (Fig. 9C). In sharp contrast, between and below these intervals (622-671 and 689-710 cm) beds are coarser (up to 18 μm), significantly thicker, and upward fining.



To estimate the depositional age of the core bottom, I developed a sedimentologic model that assumed hemipelagic sediments below the Glacier Peak tephra had the same sedimentation rate as those above the tephra. The upward-fining beds are interpreted to be event beds deposited by glacial outwash or flood events during the late Pleistocene glacial retreat (Sperazza et al. 2002; Hofmann 2005). Each upward-fining bed reflects

the waning of an episodic flood flow over a short period of time. I assigned a time interval of 1 year to each upward-fining bed to reflect the short duration of deposition.

The total thickness of most of the upward-fining beds exceeds the 1 cm step interval used in the chronologic model for the entire core. Using core photos and/or grain size data, each centimeter below the Glacier Peak tephra was examined to determine the ratio of hemipelagic/event bed deposition. Each 1 cm interval was assigned a depositional time based on the percent of hemipelagic laminae (if any) multiplied by the hemipelagic depositional rate (26.3 yrs/cm) calculated directly above the Glacier Peak tephra (see below) plus one-year for each upward-fining bed present in that interval. Based on this formula (see eq. #3 below) the chronology could be extended below the Glacier Peak tephra, although there is little basis for quantifying the uncertainty of this method.

Initial Chronology

Based on the chronological controls discussed above, depositional rates were calculated for each of the three segments of core 9P separated by the tephrae. The first portion of the core covers the interval from the top of the core to the lowest occurrence of the Mt. Mazama tephra and has a calculated depositional rate (eq. 1) of 0.45 mm/yr.

$$1) \quad (3755 \text{ mm} - 362 \text{ mm ash} + 25 \text{ mm missing at top of core}) / 7630 \text{ years or} \\ (3418 \text{ mm} / 7630 \text{ yrs} = 0.45 \text{ mm/yr}).$$

The depositional rate calculation (eq. 2) between the Mt. Mazama tephra and the Glacier Peak tephra is 0.38 mm/yr.

$$2) \quad (2335 \text{ mm} / 6125 \text{ yrs}) = 0.38 \text{ mm/yr.}$$

The last portion of the core covers from the Glacier Peak tephra to the bottom of the core. As discussed above, no solid time constraints exist below the tephra. The age model was developed based on the assumptions outlined above (eq. 3).

$$3) \quad ((1 - \% \text{hemipelagic laminae}) * 26.3 \text{ yr/cm}) + 1 \text{ year for each upward-fining bed present in the 1 cm interval.}$$

Figure 12 shows the initial chronological model based on the methods described above.

Refinement of Initial Age Model:

The initial chronology of core 9P was based solely on a limited number of date points (two-tephra dates and the $^{137}\text{Cs} / ^{210}\text{Pb}$ isotope correlation date) and was, as a result, rather crude. The initial age model provided multi-millennial scale resolution. Refining this initial chronology was necessary to tighten our resolution and place the core suite into a more useful temporal context.

Bulk Carbon Dating

To test the possibility of radiometrically dating bulk sediment within core 9P, two samples were sent to Beta Analytic, Inc. for ^{14}C AMS dating. The samples were taken close to each of the tephras so that they might provide a basis for assessing potential carbon reservoir effects. Sample FL-00-9P-III-82, taken 14 cm below the Mazama

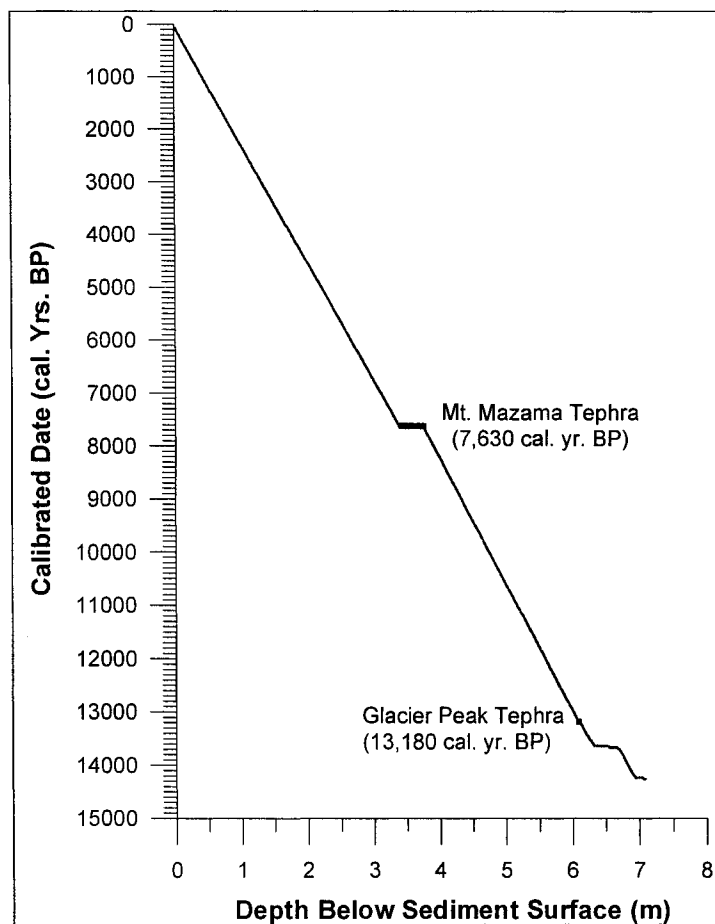


Figure 12 – Initial chronostratigraphic model based on two tephra tie points, $^{137}\text{Cs}/^{210}\text{Pb}$ dating, and sedimentary upward-fining beds.

tephra, did not yield a date due to low residual carbon; following pretreatment, the mass of recovered bulk carbon was below the one milligram AMS requirement. Sample FL-00-9P-V-74, taken 1 cm above the Glacier Peak tephra, returned a radiocarbon date of $15,720 \pm 380$ cal. yr. BP ($13,070 \pm 375$ ^{14}C BP). This bulk date is two millennia older than the Glacier Peak G tephra (Appendix A), almost certainly reflecting a significant reservoir of older carbon. According to Ron Hatfield (2002), from Beta Analytical who conducted the analysis, approximately 15% of the carbon in sample FL-00-9P-V-74 is radiometric dead (Ron Hatfield 2002, pers. comm.). Sources of the dead carbon can be

numerous and may include reworked organic material (including bits of Cenozoic coal that occurs in the East Kootenay Coal District, British Columbia in the upper Flathead River watershed (Kalkreuth 2004), detrital carbonate, authigenic carbonate, and residual carbon from the groundwater.

Whereas researchers have made adjustments for reservoir effects in radiometric dating (Benson 1993; Geyh et al. 1999) these corrections can be inaccurate and imprecise when the carbonate source is uncertain and varies with core depth. To compensate for the reservoir effect, most studies have applied a single adjustment to the entire length of the core, typically based on a modern reservoir measurements (Lund and Banerjee 1985; Benson 1993). However, this approach does not account for reservoir changes over time and thus cannot completely constrain the dating uncertainty (Benson 1993; Geyh et al. 1999; Dumond and Griffin 2002). Because of the large uncertainty involved, establishing a reservoir factor for correcting the bulk radiometric dates would not provide the desired resolution. Without the ability to quantify accurately the carbon reservoir effect for Flathead Lake, I decided to exclude the bulk dates from my initial chronology and not pursue the method further.

Paleomagnetic Secular Variation

During the initial analysis of piston cores collected from Big Arm Bay in August of 2003 (FL-03-15K, FL-03-16K, and FL-03-19K), a number of twigs of sufficient size were recovered for dating. Of these, a total of eight woody debris samples were sent to Beta Analytic for radiocarbon dating (Table 4, Appendix A). The strategy for refining the

chronology for core 9P was to correlate the carbon dates among Flathead cores FL-03-15K, FL-03-16K, and FL-03-19K and other well-dated reference lakes by way of paleomagnetic secular variation (PSV). The use of secular variation previously has been established as a dating method for marine and lacustrine sediments (Verosub et al. 1986; Hanna and Verosub 1988; Hagee and Olson 1989; Omarzai et al. 1993; Negrini et al. 2000; Benson et al. 2003). Recent studies have shown that paleomagnetic secular variation trends can be correlated across North America and globally between continents (Lund 1996; Itota et al. 1997).

Secular variation is a measure of the total magnetic field that gradually changes over time (McElhinny and McFadden 2000). The magnetic field vector can be decomposed into inclination and declination components. Declination is the measure of the azimuthal angle between the horizontal component and geographic north, while inclination is the vertical or dip component that is measured as the angle down from the horizontal component (Butler 1992). These components record both the magnetic dipole (similar to a magnetic bar) and nondipole fields (Tauxe 1998). The geomagnetic pole, which is the surface projection of the dipole part of the magnetic field can be derived from secular variation data obtained from multiple localities (Butler 1992; McElhinny and McFadden 2000). Complete discussions of magnetic fields can be found in Thompson and Oldfield (1986), Butler (1992), Tauxe (1998), McElhinny and McFadden (2000), and Campbell (2003) and are not reviewed here.

Secular variation measurements on U-channels accurately measure low frequency changes in inclination and declination with spatial resolution of 3-5 cm on continuous sequences of core sediments (Nagy and Valet 1993). Sediment U-channels were extracted from the archive half of cores FL-00-9P, FL-03-15K, FL-03-16K, and FL-03-19K and sent to the paleomagnetism laboratory at the University of California-Davis for magnetostratigraphic measurements on a 2-G Enterprises Model 755 Cryogenic Magnetometer. Timmerman (2005) collected inclination and declination data for each of the U-channels and utilized vector and principle component analyses to determine that the 30mT demagnetization step accurately represented preserved paleomagnetic vectors in the Flathead Lake cores and could be used for secular variation correlations. A detailed discussion on the methods used to collect the PSV data are presented in Timmerman (2005) and will not be repeated here.

In addition to the paleomagnetic data collected for the cores from Flathead Lake, I used Holocene and latest Pleistocene secular variation data from two well-dated reference lakes: St. Croix Lake (Lund and Banerjee 1985) and Fish Lake (Verosub et al. 1986; Verosub 1988). I also considered a reinterpreted data set for Fish Lake (Steve Lund, 2004, written. comm.).

The methodology for correlating the paleomagnetic secular variation entails visually correlating the inclination and declination records of the reference records to those of the core to be dated. Software aids for the correlation of time-series data, such as secular

Lake/Core	Depth (mbsf)	¹⁴ C Yrs. BP	cal. yr. BP	Source
FL-00-9P	3.78 ¹	6845 ± 85	7630 ± 80	Zdanowicz et al. 1999
	6.74 ²	13070 ± 40	15720 ± 380	Beta-176803
	6.76 ³	11200 ± 120	13180 ± 120	Hallett et al. 2001
FL-03-15K	0.44	2880 ± 50	2980 ± 145	Beta-184123
	0.90 ¹	6845 ± 85	7630 ± 80	Zdanowicz et al. 1999
	0.95	6890 ± 40	7700 ± 65	Beta-183410
	2.22	8780 ± 40	9760 ± 125	Beta-183411
	3.53 ³	11200 ± 120	13180 ± 120	Hallett et al. 2001
FL-03-16K	0.44	4450 ± 40	5040 ± 205	Beta-183412
	2.70 ¹	6845 ± 85	7630 ± 80	Zdanowicz et al. 1999
	3.39	7900 ± 40	8650 ± 80	Beta-183413
	4.54	9040 ± 50	10210 ± 40	Beta-183414
	5.99	10340 ± 50	12220 ± 95	Beta-183415
	6.54 ³	11200 ± 120	13180 ± 120	Hallett et al. 2001
FL-03-19K	1.47 ¹	6845 ± 85	7630 ± 80	Zdanowicz et al. 1999
	2.97 ³	11200 ± 120	13180 ± 120	Hallett et al. 2001
	4.96	12230 ± 50	14150 ± 145	Beta-183416
Lake St Croix ⁴	0.72	100 ± 125	75 ± 100	Lund and Banerjee 1985
	1.95	416 ± 72	505 ± 75	Lund and Banerjee 1985
	3.65	802 ± 72	710 ± 80	Lund and Banerjee 1985
	5.80	1096 ± 72	985 ± 75	Lund and Banerjee 1985
	8.9	1944 ± 78	1895 ± 85	Lund and Banerjee 1985
	11.9	2928 ± 92	2930 ± 100	Lund and Banerjee 1985
	14.9	4683 ± 87	5445 ± 95	Lund and Banerjee 1985
	16.75	6964 ± 107	7790 ± 115	Lund and Banerjee 1985
	18.78	9634 ± 129	11005 ± 135	Lund and Banerjee 1985

Table 4 – Radiometric ¹⁴C dates for cores in this study. Calibration to calendar years BP based on InterCal98 model (Stuiver et al. 1998) and calculated using OxCal version 3.9 for non Beta data. Calibrated date uncertainty based on 1 sigma results. Notes: ¹-Mt. Mazama Crater Lake tephra dates based on (Zdanowicz et al. 1999). ²-Bulk sediment carbon date excluded from chronology due to reservoir effect. ³-Glacier Peak G tephra date based on (Hallett et al. 2001). ⁴-Radiometric dates from Lund and Banerjee (1985), calendar year calibration based on OxCal version 3.9. Mbsf = meters below sediment surface.

variation, are very few in number. In his preliminary secular variation correlation among some of the Flathead Lake cores, Timmerman (2005) used AnalySeries version 1.2 (e.g., Prell et al. 1986). However, AnalySeries still requires the operator to manually identify visual correlations and only simplifies the correlation process by stretching or compressing PSV data between specific date points. In this study, I elected to correlate tie points and graphically shift PSV curves manually on an Excel spreadsheet. I accomplished visual correlations with the aid of Grapher software version 5. Specifically, I correlated a set of tie points between the reference cores from Fish and St. Croix lakes and assigned the appropriate resulting date to the corresponding 1 cm depth interval in the Flathead Lake core. I then calculated a sedimentation rate for each segment between the dated tie points and constructed a new chronostratigraphic model that assigns a date to each centimeter of the core.

Declination Rotation

The analysis of paleomagnetic declination measurements required some rotation about a vertical axis prior to correlation, so that all of the individual sections within a single core are consistently oriented. The misalignment of declination data from individual core sections can be caused by several variables, including: 1) rotation of sediment as it enters the core barrel during the drive, 2) inconsistent orientation of each core segment during the splitting process, 3) insertion of the U-channel into the core at an angle that is non-orthogonal to the split core surface, and 4) partial rotation of sediment that is not retained in the U-channel and must be extracted from the core section and placed into the U-channel by hand.

Three methods are available for aligning declination data of adjacent core sections: 1) point matching – rotation of a core section so that the two data points across the core section gap have the same declination value. This method assumes that no large declination change has occurred across the gap. 2) trend matching – the trend of declination data from one core section is extended across the section gap to the adjacent core section. This method assumes that a trend, which may be only derived from two points, can be projected across the gap. 3) cross-correlation – using a replicate core to reconstruct the gap from the matching declination record. This method assumes that the core segments have not rotated (see below) and a replicate core has been recovered. Since cross-correlation does not make any assumptions regarding the actual variation of the declination, it affords the best reconstruction across the section gaps (Lund and Banerjee 1985). Because replicate piston cores were not recovered in Flathead Lake, however, it was necessary to use either the point matching or trend matching techniques.

During initially processing core 9P, no efforts were made to maintain a consistent orientation while splitting the core sections horizontally. This was not the case when processing cores from 2003, which were split with the orientation line (a hand drawn line down the entire length of the polypropylene core barrel) facing up. Thus it was necessary to rotate each of the core sections from 9P about a vertical axis in order to maintain a consistent declination record. In this study, the point matching method accomplished declination rotations between core sections.

Rotation of sediments within the core liner during the coring operation has been mentioned as a possible problem but is a poorly understood process and difficult to recognize (e.g., Breckenridge et al. 2004). During the recovery of sediments from Flathead Lake, several cores reached the lake surface either wrapped in the trigger wire or showing a spiral pattern on the mud coating on the outside of the metal core barrel. Both occurrences suggest the core barrels had rotated, likely during removal of the core from the bottom after the drive.

In order to investigate potential disturbances or rotation during the coring process, I visually inspected the sediments, core photographs, and X-radiographs from cores 9P, 15K, 16K, and 19K. These inspections suggested the presence of one disturbance, in core 19K (400-490 cm) that may have occurred during coring, although a naturally occurring disturbance during sediment deposition could not be ruled out. In this core section, horizontally-stratified glacial varves show a progressive disturbance starting with intact but moderately-dipping beds that grade into steeply-dipping, deformed beds and end with beds that appear to be sub-vertical in the upper ~40 cm of the section. The disturbed structures are present in both photographs and X-radiographs.

As part of this analysis, I examined photographs taken during the coring operation for clues that might suggest core barrel rotation during recovery. Photos showing the core barrel with the mud coating clearly display both straight and spiral lineations (Fig. 13). Based on the limited number of photographs available, it appears that the spiral lineations crosscut the straight lineations. Rotation of the core barrel during recovery does occur, as

indicated by the tangled trigger cable that is secured above the core barrel during the decent. However, rotation during the core drive is likely to be insignificant as descent of the core barrel is paused about three meters above the sediment-water interface, allowing the Kullenberg coring apparatus to stabilize prior to release and penetration. The spiral lineations are of low rotational frequency, usually one to two rotations per core length (~10 m), suggesting small rotational stress as tension is reapplied to the twisted cable. While these investigations suggest core rotation is a post drive recovery process, these processes remain poorly understood and not well documented.

To further investigate potential sediment rotation resulting from the coring, I examined the magnitude of corrective declination rotation for the 2000 and 2003 PSV data sets from Flathead Lake. The idea behind this investigation is that cores processed with an effort to maintain consistent orientation should show smaller declination corrections than cores that were split without attempts to maintain a consistent orientation. Despite every effort to keep sampling procedures consistent down the length of an entire core, some orientation variability is still expected and reasonable due to the following: 1) the orientation line is hand drawn on a curved core liner surface and is not always perfectly straight. 2) During the core splitting process, the orientation line is positioned by eye to be facing directly up. However, no alignment marks exist on the cutting apparatus, so it is likely that up to five degrees of uncertainty in core orientation is introduced in this manner. 3) It is possible that minor rotation of the sediment within the split core liner can occur during handling of the core. 4) A non-orthogonal insertion of the U-channel into

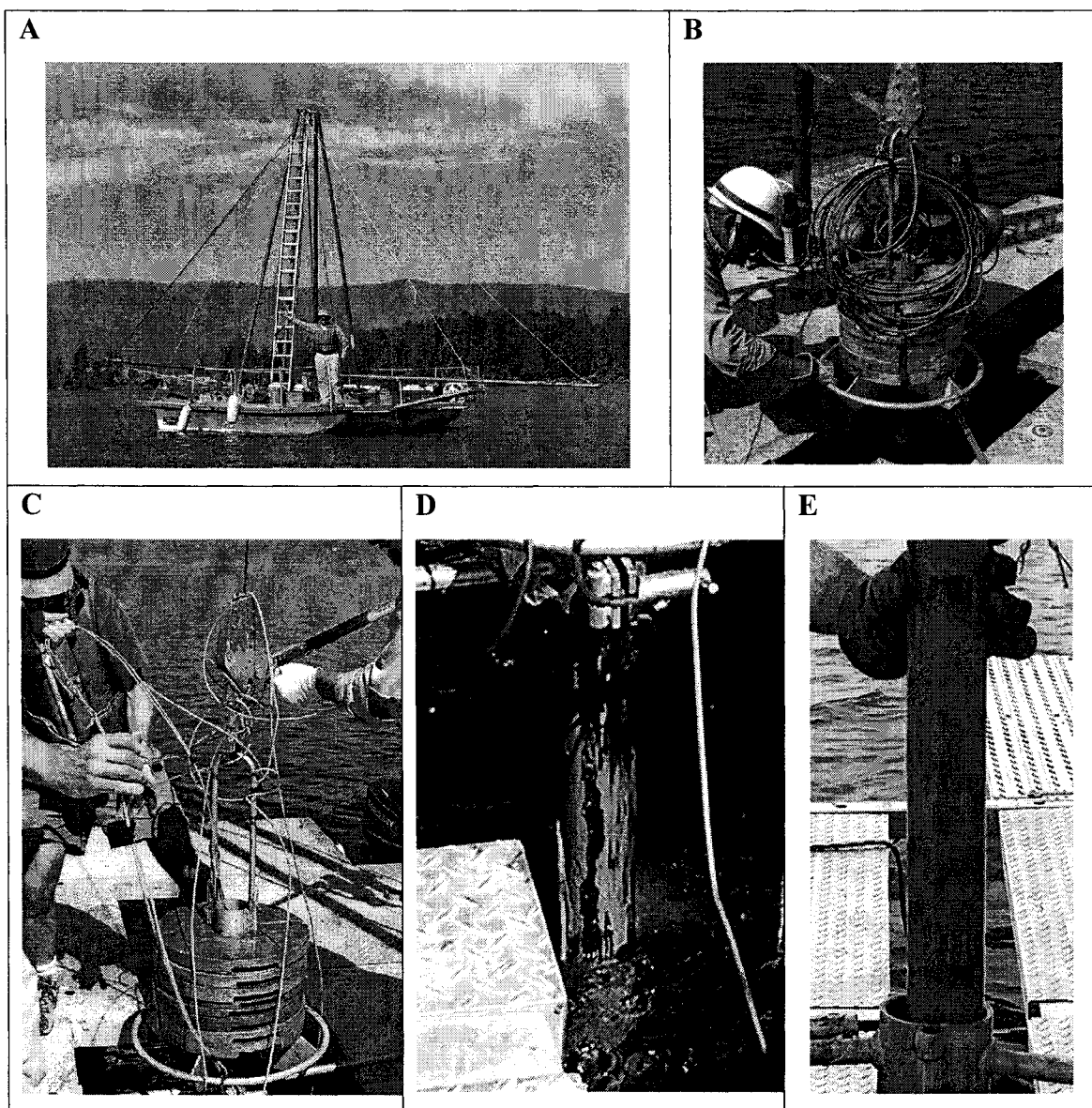


Figure 13 – Coring photos. A) Coring platform from the University of Minnesota Limnological Research Center. B) Trigger wire in coiled position prior to core drive. C) Trigger wire after core drive tangled and wrapped around weight stand. D and E) Sample photos of predominately straight lineations in mud covering the exterior of steel core barrel. These two samples show straight lineations are consistent over the length of the core. D) Photo of straight lineations at the top of the core barrel (note weight stand at top of photo) and lineations shown in the middle of another core barrel (E).

the split core surface might introduce slight rotational variations among individually sampled core sections. 5) It is difficult to maintain the surface of the split core half in a perfectly horizontal orientation during the sub-sampling process, due to the curvature and flexure of the core liner and the fact that significant pressure must be applied to the U-channel to drive it into compacted mud of the core. 6) Recovery of the tephras and terminal few centimeters of each core section occasionally would not stay firmly inserted in the U-channel, requiring placement by hand.

A graphical analysis of the declination data for the entire suite of Flathead cores (Fig. 14) shows a higher degree of corrective rotation for the 2000 cores than the cores processed in 2003. Additionally, with only a couple of exceptions, declination records did not contain any sharp shifts that may indicate a rotation within the core section. The three exceptions to this are in core 22K sections I & III and section IV of core 8P. Each of these mid-section shifts are contained within the top or bottom 20 cm of the section and are likely related to displacement of sediments during U-channel acquisition.

Additionally, rotation during coring operation would likely produce a ‘smeared’ appearance in the declination data instead of a quantum jump or some sort of step-function; this ‘smearing’ effect is not observed at any of these mid-section shifts. A single exception appears to exist in the Mt. Mazama interval of core 9P, where the PSV declination record is ‘smeared’, suggesting rotation of the sediments during slumping.

Timmerman (2005) performed declination rotation for all cores utilized in this study. In core 9P the declination poles of sections II-V were normalized to section I by adding or

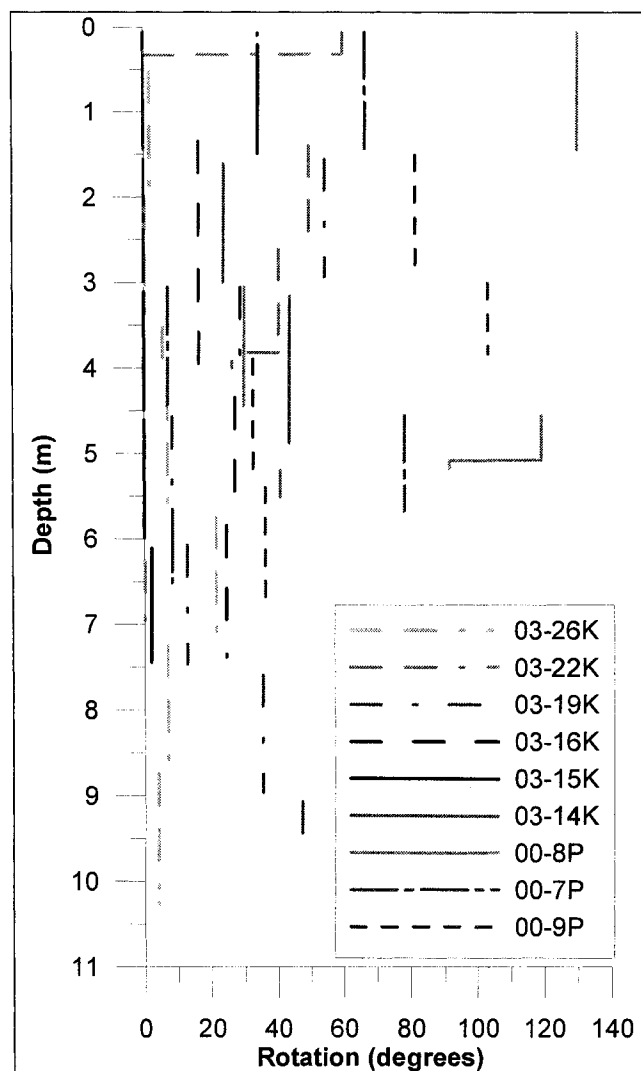


Figure 14 – Rotation of paleomagnetic declination for Flathead cores used in dating correlation. Lines show amount of core rotation by section. The legend ‘00’ identifies cores recovered in 2000 and ‘03’ for cores from 2003.

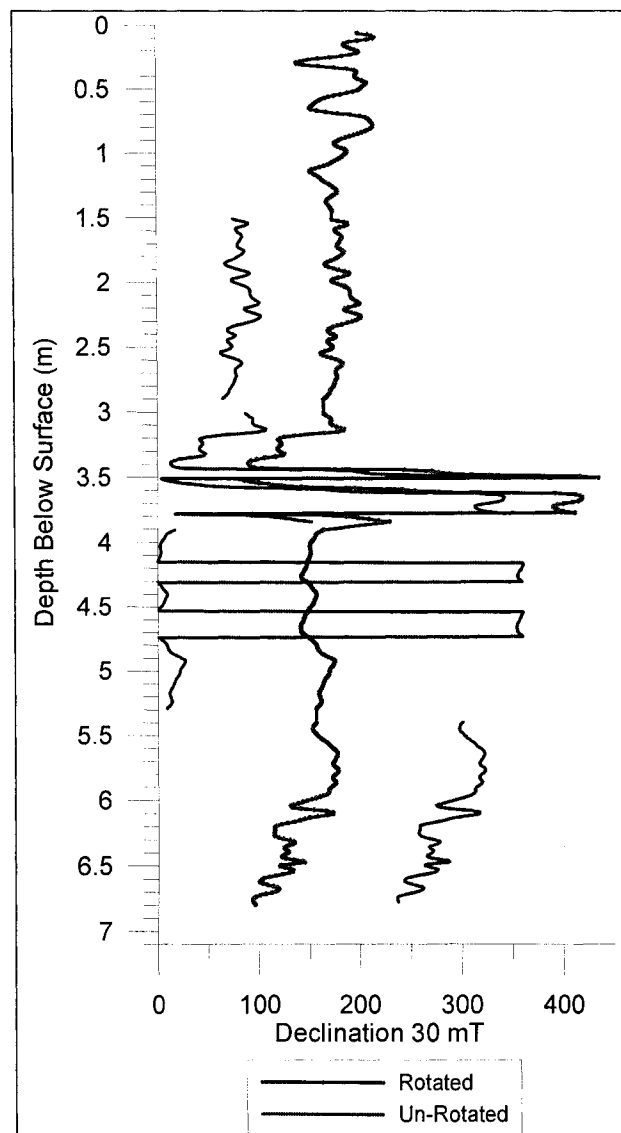


Figure 15 – Rotation of paleomagnetic declination for sections of core 9P. Rotated line shows data points (red). Un-rotated line indicates amounts of rotation applied to core 9P. Mazama tephra (~340-385 cm) shows internal rotation, assumed to reflect post-depositional reworking or slumping. This section was not rotated but assigned the same age of deposition. Two sharp shifts at ~415 and ~453 cm are graphical artifacts of plotting spherical data in two dimensions.

subtracting the appropriate number of degrees to align the uppermost declination measurement with the bottom of the preceding section (Fig. 15). Section III was complicated by three repeated sections of the Mt. Mazama tephra, each of which likely occurred during syn/post-depositional reworking and/or slumping, as discussed above. In section IV, two sharp shifts are present at ~415 and ~453 cm (Fig. 15). These are graphical artifacts of spherical declination data that vary about a continuous 360-degree pole, but are being graphically displayed on a two-dimensional plot.

Correlation Data

Two types of reference data were available for PSV correlation work in this study: data within Flathead Lake and data external to the lake basin. Data from within the lake, particularly cores FL-03-15K, FL-03-16K, and FL-03-19K, were preferred for correlation because I expected them to contain the fewest significant angular PSV variations due to location differences and because local magnetic anomalies might be expected to affect each of the Flathead Lake cores in the same manner.

Data external to the lake basin was obtained for two well-dated PSV reference lakes, Lake St. Croix (Lund and Banerjee 1985, Steve Lund, 2004, written comm.), and Fish Lake (Verosub et al. 1986, Steve Lund, 2004, written. comm., Kenneth Verosub, 2004, written. comm.). The actual ^{14}C date points for Lake St. Croix were published (Table 4, Lund and Banerjee 1985), however the ^{14}C data for Fish Lake have not been published and are apparently lost (Kenneth Verosub, 2004, pers. comm.). The original data for Fish Lake have been reinterpreted by Lund (1996). Additionally, Lund (1996) identified a set

of major peaks and valleys in Holocene declination and inclination records from eight lacustrine settings and one ARCMAG record forming a traverse across North America. These nine localities, which include Lake St. Croix and Fish Lake, can be used as PSV reference sections for correlation with PSV records from new localities, such as Flathead Lake (Appendix B). Using the nine independently dated PSV records, Lund (1996) calculated the average date and dating uncertainty for each of the major inclination and declination tie points.

Graphical Correlations

The goal behind the PSV technique is to use the shape of the PSV curves to correlate among cores within the Flathead Lake (FHL) basin and, by extension, correlate dated stratigraphic levels among cores to erect a lake wide chronostratigraphy. An important component of this work is to correlate dated portions of the reference lake cores outside the FHL basin with the PSV signal from Flathead Lake cores. Declination and inclination records are considered to be separate data sets; correlations of each were made independently (Omarzai et al. 1993; Breckenridge et al. 2004; Rolph et al. 2004).

All PSV data first were considered plotted against core depth (Figs. 16 & 17). However, using PSV data with a depth scale created visual correlation problems. First, no depth data was available for the original Fish Lake data set (Verosub et al. 1986). Second, differences in sediment accumulation rates vertically distorted the PSV records, even within Flathead Lake. Accordingly, all PSV records were plotted based on age scale prior to initial correlation, using the tephras as the initial tie points among the Flathead

cores and with the PSV age models for Fish Lake (which includes the Mazama tephra) and Lake St. Croix (Figs. 18 & 19). Age model plots in Figures 18 & 19 for the Flathead cores use a simple linear interpolation between the dated points (tephra or ^{14}C). Selected dates from published age models for Lake St. Croix (Lund and Banerjee 1985) and the revised Fish Lake data (Lund 1996) were converted from ^{14}C years to cal. yr. BP with OxCal 3.9 calibration software. The inclination and declination data were then plotted based on the calibrated dates for use in the graphical correlations.

Inclination and declination records of core 9P were correlated separately using the North American tie points identified by Lund (1996) for Fish Lake and Lake St. Croix reference lakes and the ^{14}C data from the Flathead cores. I started by plotting all the actual date points (tephra or radiometric) on each core, along with data uncertainty values (Figs. 20 & 21, and Table 4). Core FL-03-19K (not shown) was of little value in this correlation, because it contains only one direct radiometric date that, based on visual correlation of up-ward fining beds, is older than the bottom of core 9P. Figures 18 to 23 only plot the original Fish Lake data set because the PSV variations are the same for either of the two slightly different age models (Verosub et al. 1986; Lund 1996).

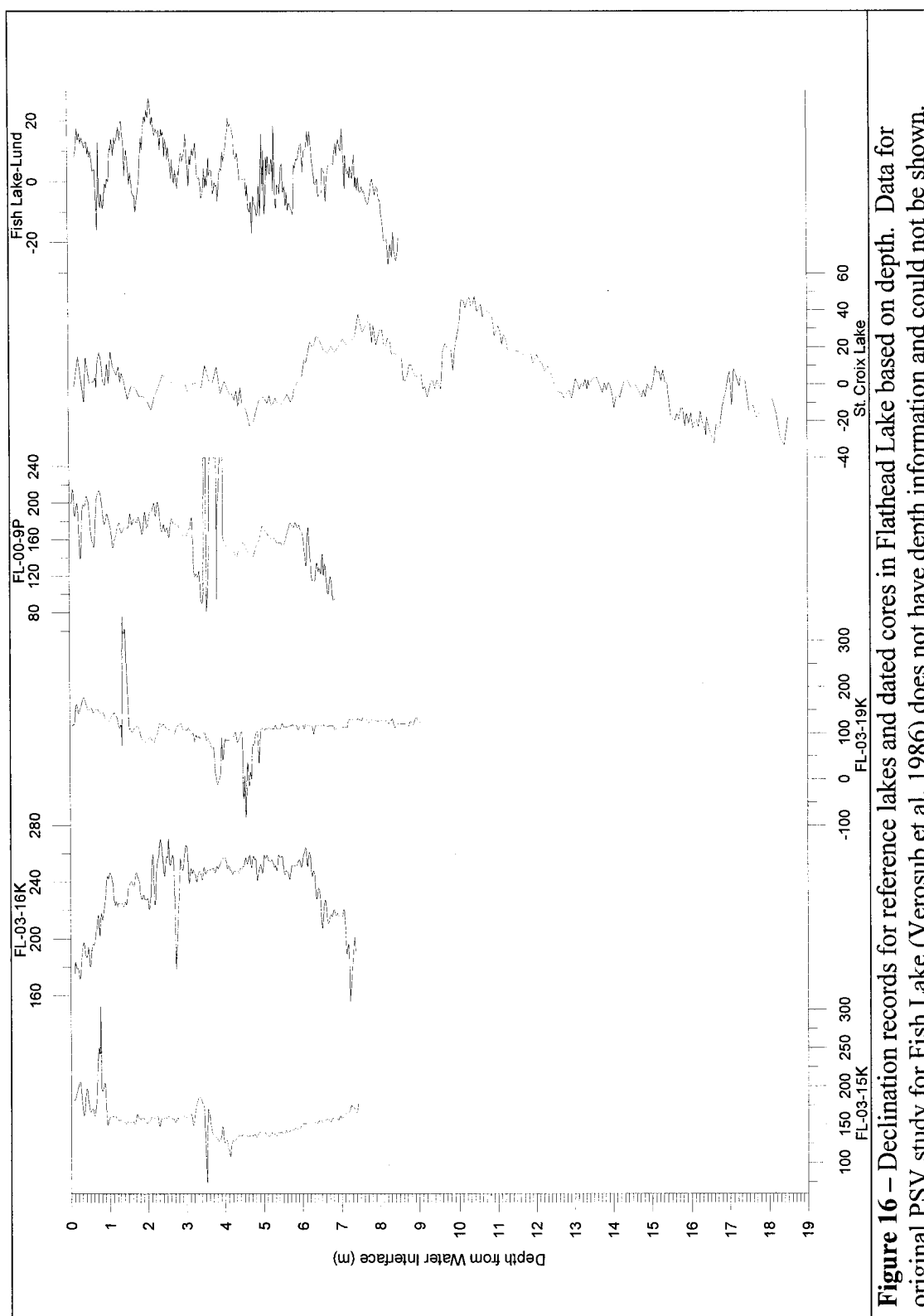
As a second step, I used the major tie points identified by Lund (1996) for Fish Lake and Lake St. Croix to correlate these records with core 9P. Most of the tie points identified by Lund (1996) could be reasonably identified; however a few were less obvious because they were not located on a maximum or minimum within the time-series data set (Figs. 22 & 23). Less convincing tie points were not utilized in this study. Figures 22 & 23

show that the average dates calculated by Lund (1996) contain a much higher uncertainty than the potential correlation points from the specific reference lakes (see uncertainty discussion below).

Based on this visual correlation approach, I was able to identify 15 separate tie points for the inclination curve and 16 tie points for declination curve (Figs. 22 & 23, and Table 5). The inclination and declination tie points were selected independently and thus provide a means of checking the reasonability of each correlation. Using a simple linear fit function between the declination and inclination-based tie points, age models were created and plotted (Fig. 24). The independent inclination and declination age models have a high linear correlation, $R^2 =$ of 0.994 and a p-value = <0.0001.

Tie Point Uncertainty

The strength of PSV correlation as a chronostratigraphic tool is limited by the researcher's ability to identify tie points between the record of interest and a reference data set. Even software tools designed to aid the correlations, such as Analyseries, relies on manual selection of tie points. Efforts by Lund (1996) to establish major tie points for the North American Holocene PSV record are the first step towards establishing a uniform methodology with constrained uncertainty. One component of PSV uncertainty is the uncertainty introduced from different operators locating the tie points based upon visual inspection. To measure this uncertainty, I asked three other individuals to pick the declination tie points in core 9P based on the data in Figure 21.



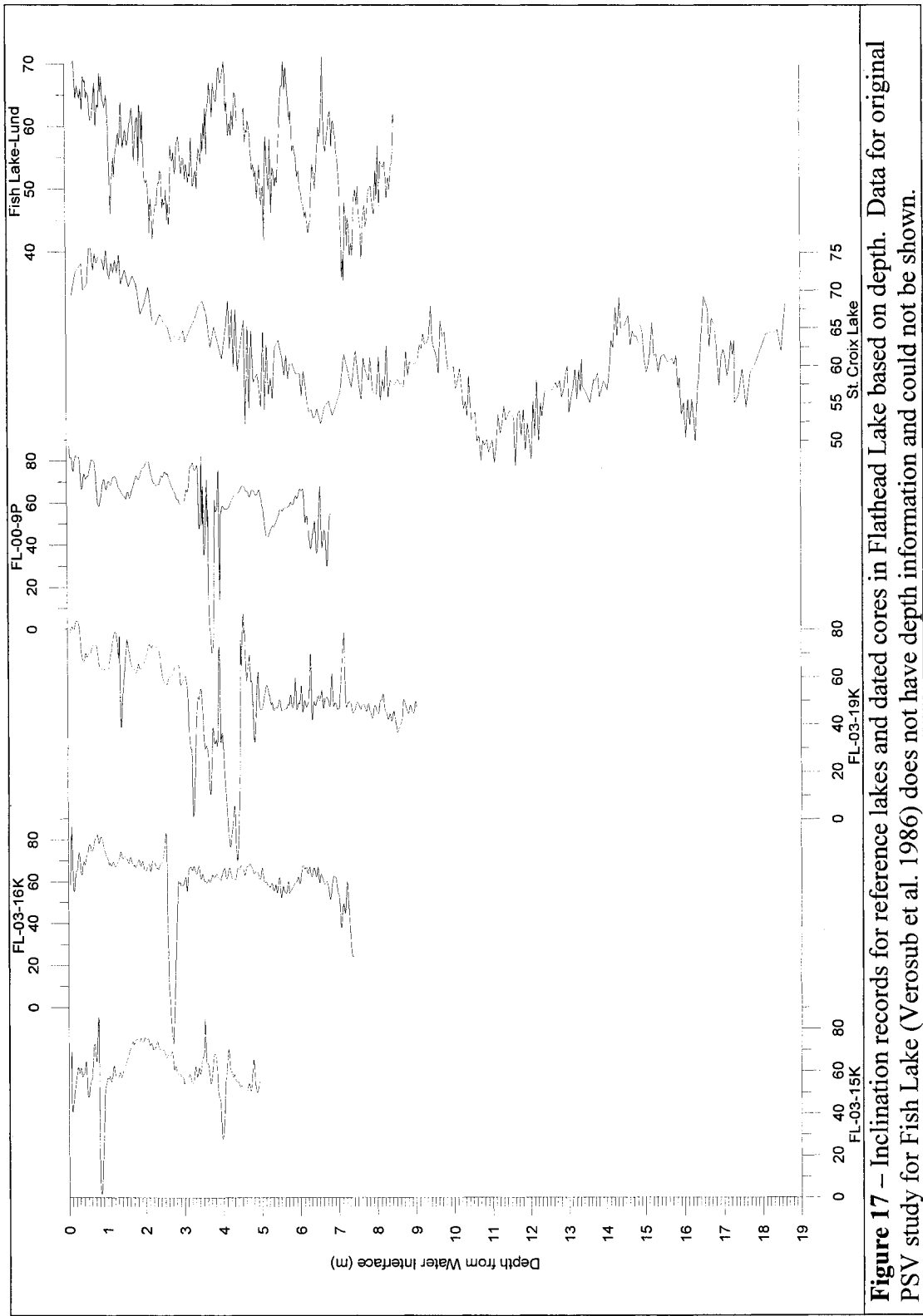


Figure 17 – Inclination records for reference lakes and dated cores in Flathead Lake based on depth. Data for original PSV study for Fish Lake (Verosub et al. 1986) does not have depth information and could not be shown.

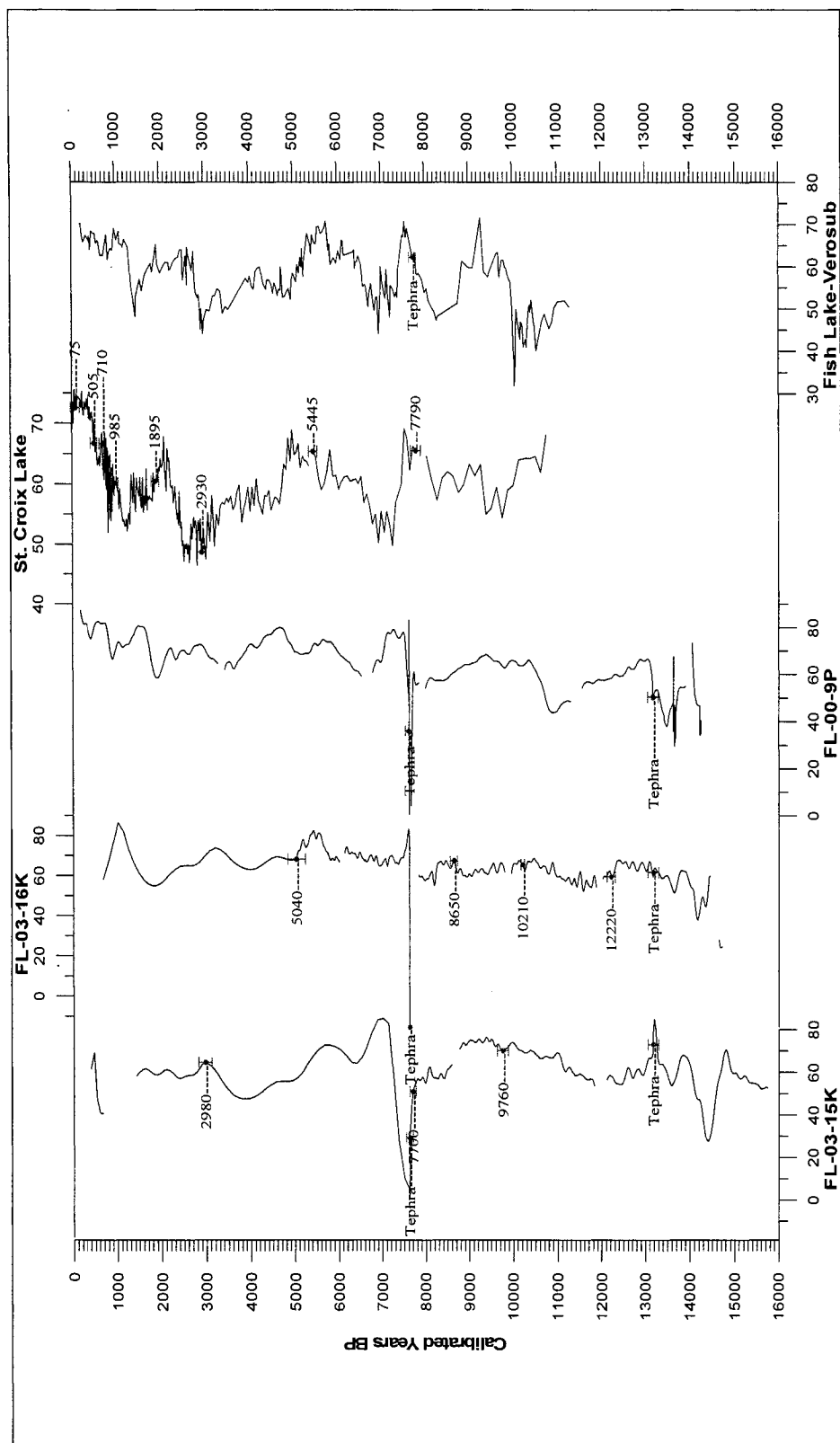


Figure 18 – Inclination data plotted based on calibrated years BP. Cores are plotted using a simple interpolation model (linear or polynomial) between the shown tie points, as described in the text. Shown for each of the cores are the actual dated points (tephra or radiometric), with uncertainty bars. Lowermost part of core FL-03-15K is truncated on this plot. Gaps in data sets are at core section breaks.

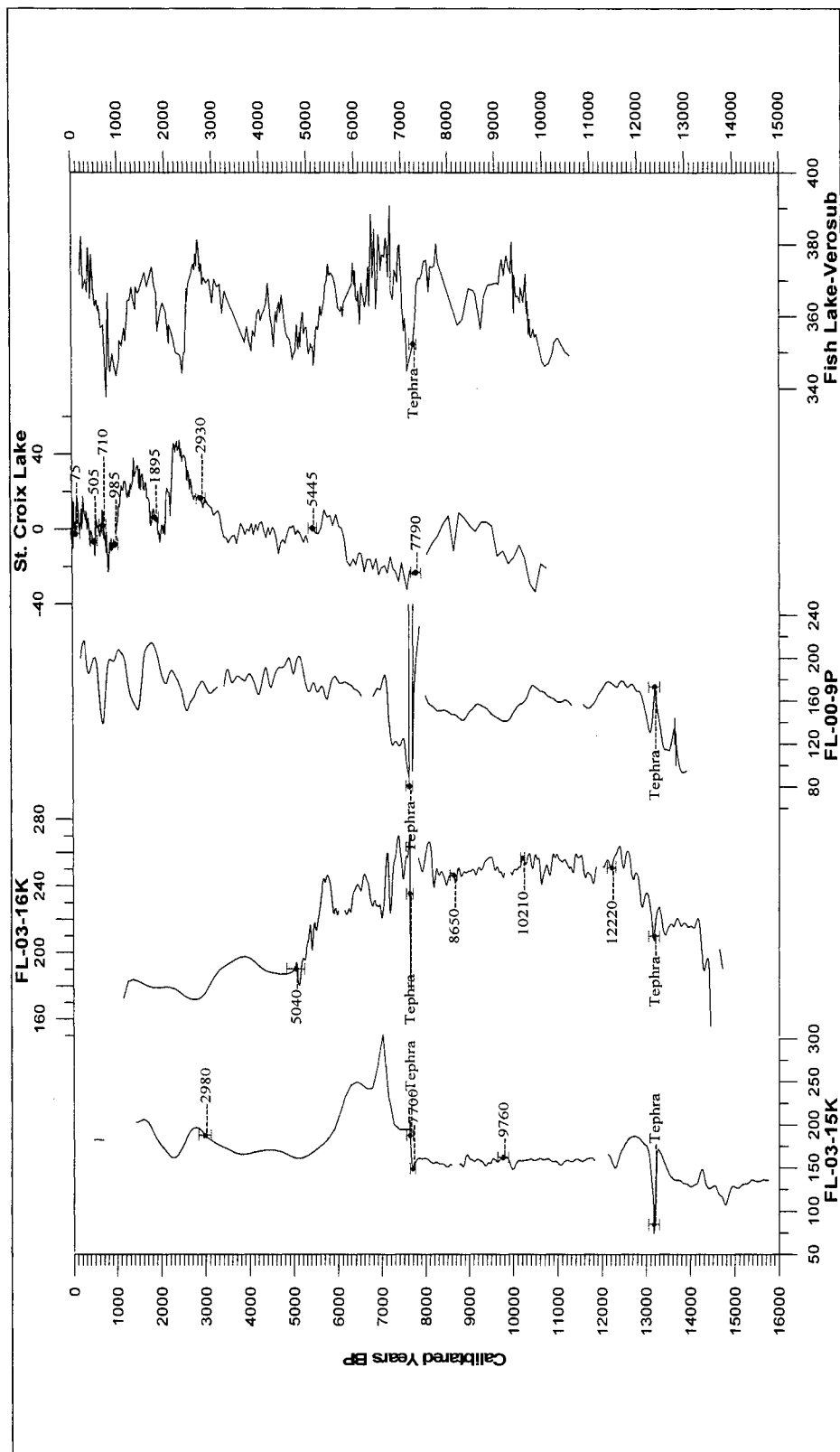


Figure 19 – Declination data plotted based on calibrated years BP. Cores are plotted using a simple interpolation model (linear or polynomial) between the tie points shown, as described in the text. Shown for each of the cores are the actual dated points (tephra or radiometric), with uncertainty bars. Lowermost part of core FL-03-15K is truncated on this plot. Gaps in data sets are at core section breaks.

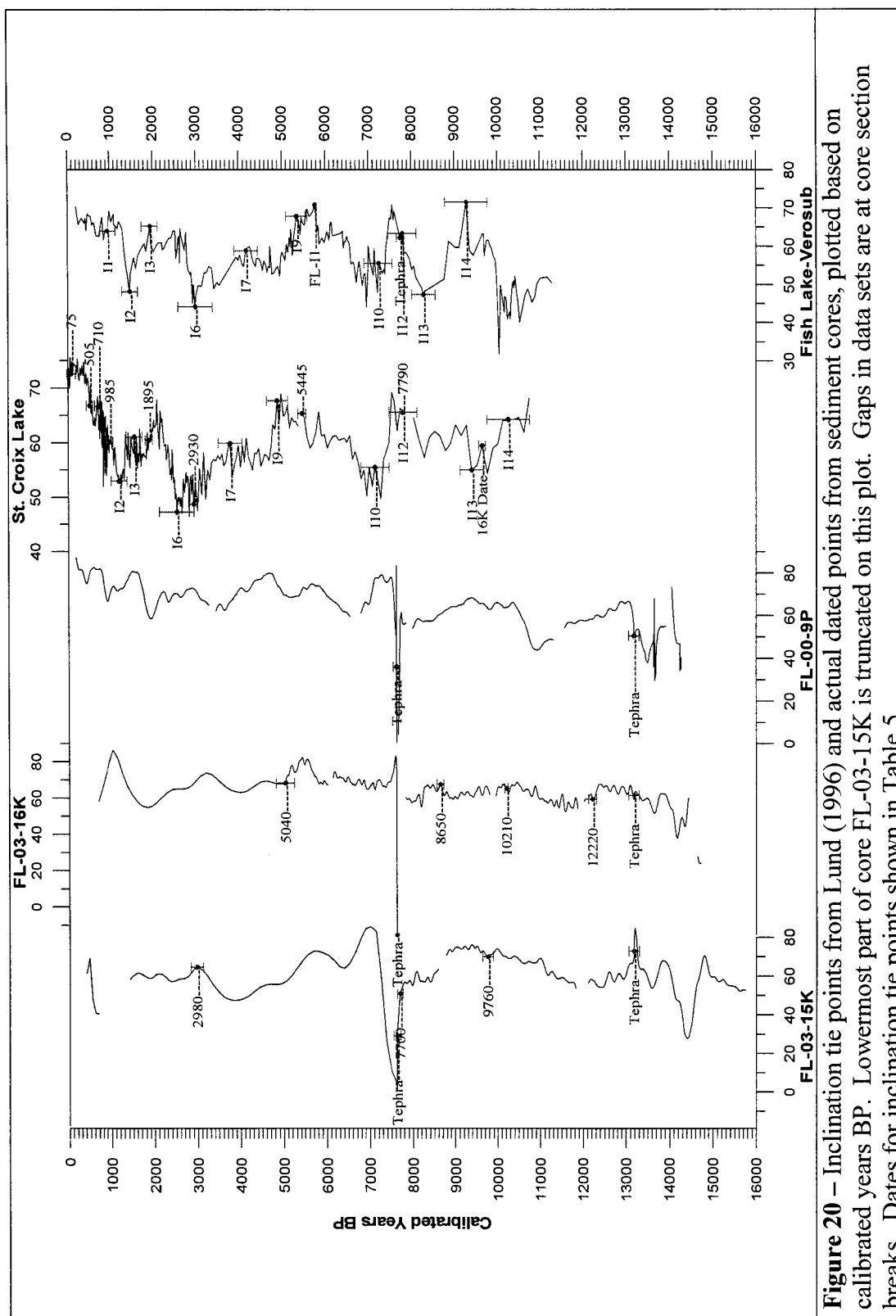


Figure 20 – Inclination tie points from Lund (1996) and actual dated points from sediment cores, plotted based on calibrated years BP. Lowermost part of core FL-03-15K is truncated on this plot. Gaps in data sets are at core section breaks. Dates for inclination tie points shown in Table 5.

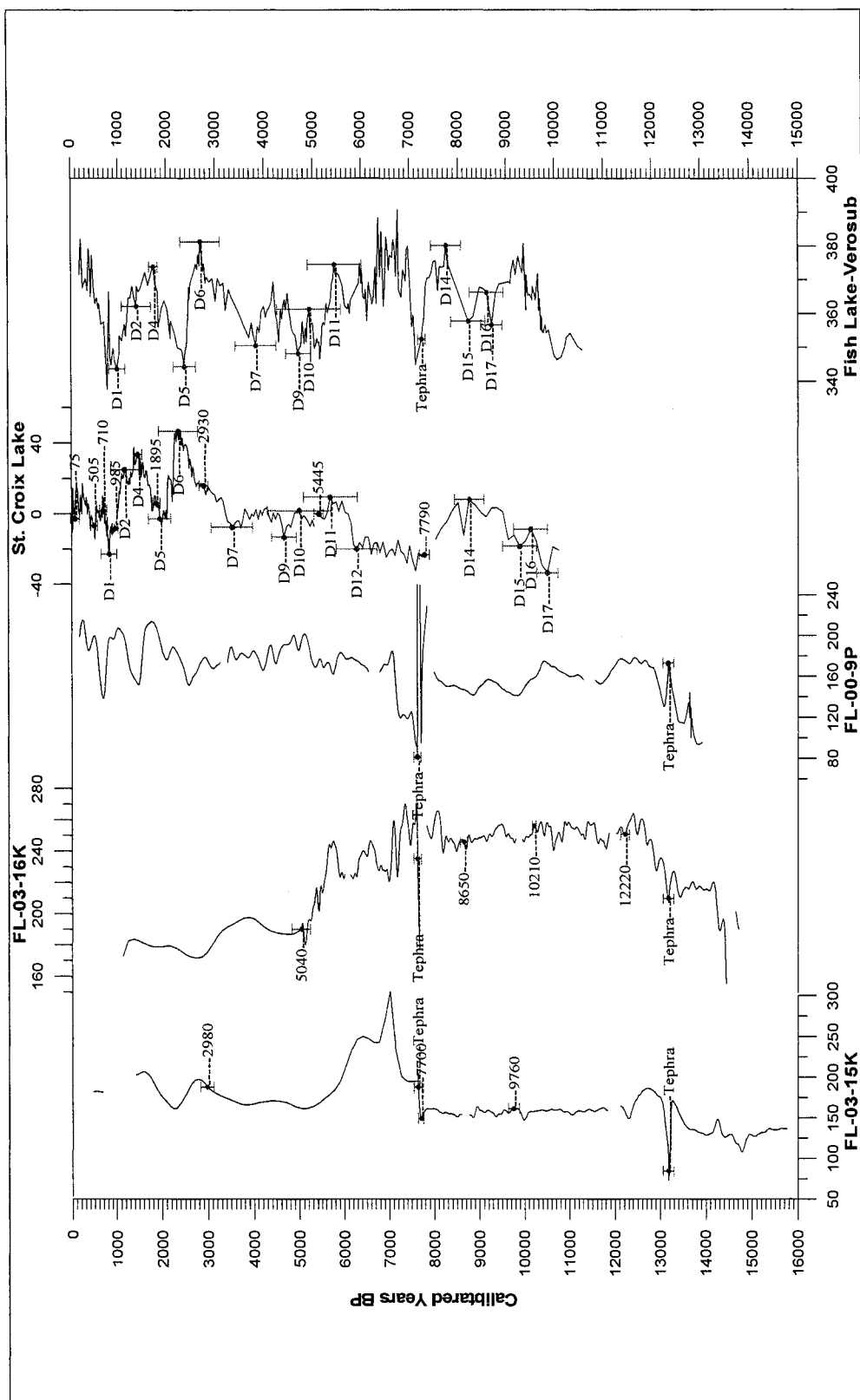


Figure 21 – Declination tie points from Lund (1996) and actual dated points from sediment cores plotted based on calibrated years BP. Lowermost part of core FL-03-15K is truncated on this plot. Gaps in data sets are at core section breaks. Dates for declination tie points shown in Table 5.

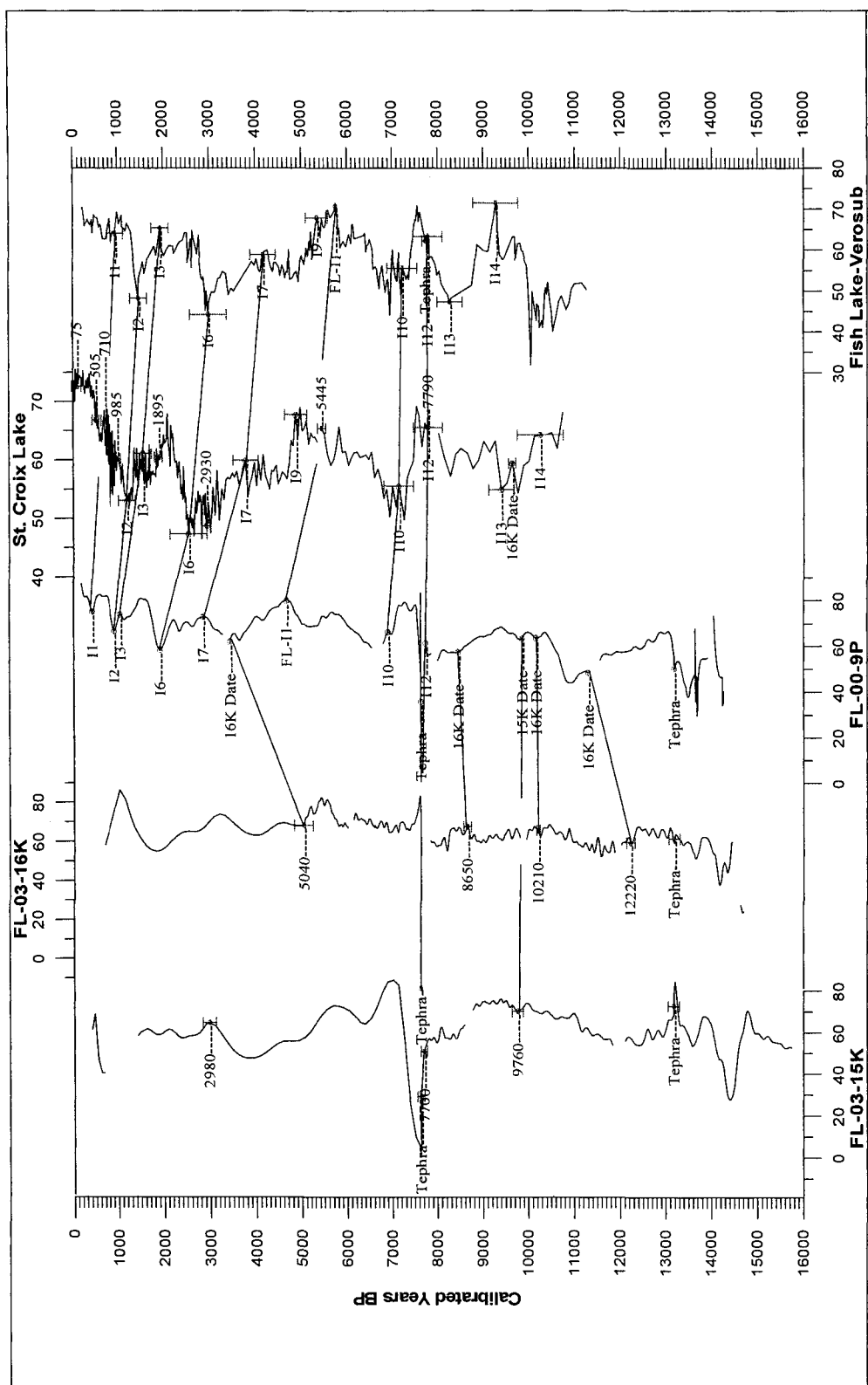
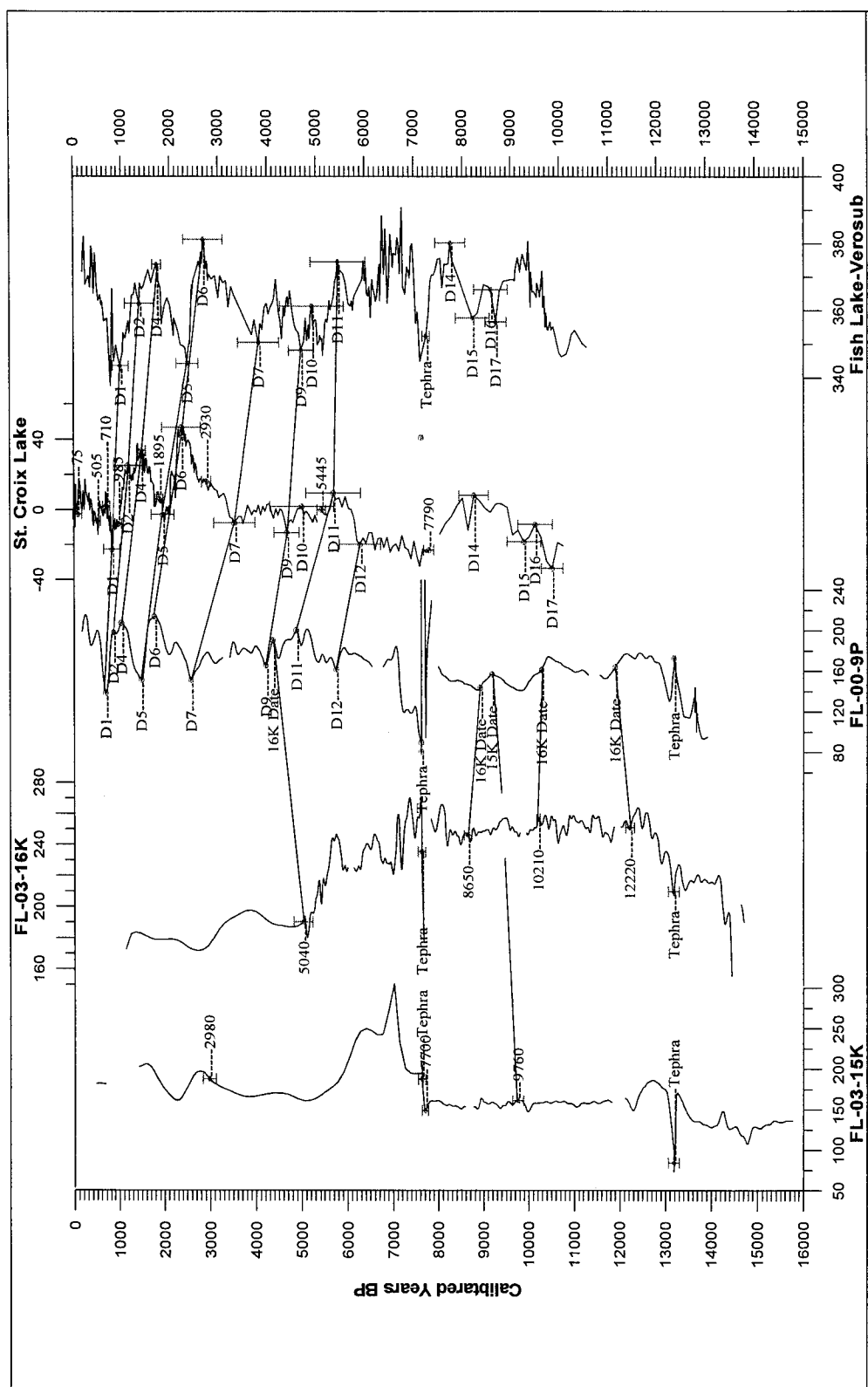
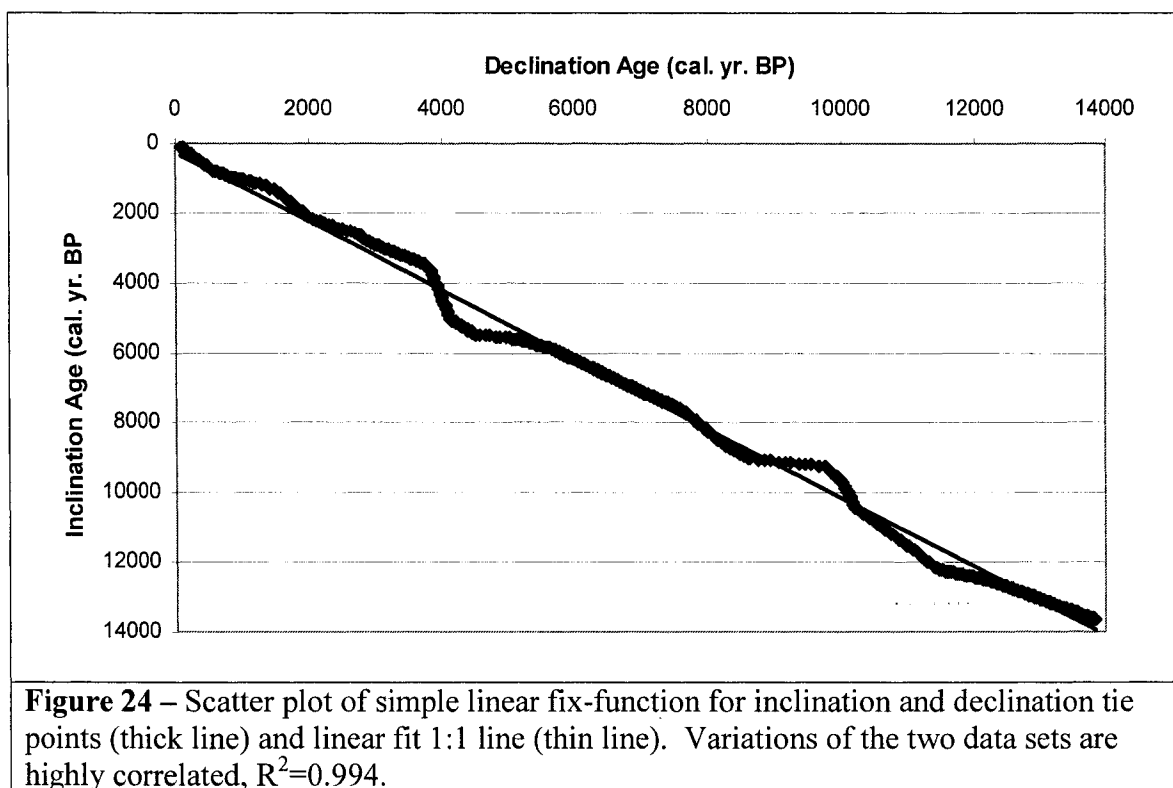


Figure 22 – Correlation of inclination tie points from reference lakes and Flathead cores. Connecting lines show correlation used in core 9P chronostratigraphic model. Core 9P inclination curve has not been adjusted to reflect correlations. Lowermost part of core FL-03-15K is truncated on this plot. Gaps in data sets are at core section breaks.



Tie Point		Average Age ¹⁴ C Yrs. BP	Calibrated Age Cal. Yrs. BP
Inclination	1	870 ± 180	780 ± 185
	2	1250 ± 180	1190 ± 185
	3	1555 ± 180	1420 ± 185
	6	2735 ± 380	2840 ± 400
	7	3415 ± 250	3680 ± 275
	16K	4450 ± 40	5040 ± 205
	FL-I1	4500 ± 250	5600 ± 250
	10	6230 ± 300	7190 ± 325
	Mazama	6845 ± 85	7630 ± 80
	12	6990 ± 300	7795 ± 325
	16K	7900 ± 40	8650 ± 80
	15K	8780 ± 40	9760 ± 125
	16K	9040 ± 50	10210 ± 40
	16K	10340 ± 50	12220 ± 95
	Glacier Peak	11200 ± 120	13180 ± 120
Declination	1	965 ± 180	915 ± 185
	2	1295 ± 270	1215 ± 325
	4	1670 ± 60	1550 ± 95
	5	2060 ± 180	2000 ± 245
	6	2530 ± 300	2730 ± 425
	7	3435 ± 320	3710 ± 450
	9	4025 ± 270	4515 ± 275
	16K	4450 ± 40	5040 ± 205
	11	4925 ± 380	5675 ± 600
	12	5485 ± 360	6295 ± 450
	Mazama	6845 ± 85	7630 ± 80
	16K	7900 ± 40	8650 ± 80
	15K	8780 ± 40	9760 ± 125
	16K	9040 ± 50	10210 ± 40
	16K	10340 ± 50	12220 ± 95
	Glacier Peak	11200 ± 120	13180 ± 120

Table 5 – Tie point data, based on correlations in Figures 18 to 23. Average ages taken from Lund (1996) showing date and uncertainty from eight well dated North American lakes and one ARCMAG locality (Appendix B). L-I1 calculated based on a Fish Lake curve date. Data in table only includes tie points used in this study. Radiometric calibration to cal. yr. BP from OxCal 3.9, except Flathead radiometric dates calibrated by Beta using Intercal v4.



I compared tie point placement between core 9P and Lund's (1996) North American Holocene PSV data set from the three independent geoscientists against my own picks. If the tie point placement was different than my placement, I used a simple linear model to calculate a date for the tie point interval. The uncertainty of each date was calculated as the percent difference among all of the tie points selected by all study participants. The average uncertainty for all 14 tie points (tephras were not included) was 3.6%. All four individuals identically selected the stratigraphic locations of seven of the nine major

tie points identified by Lund (1996) in the core 9P data set. In contrast, none of the five Flathead carbon dates were identically placed by all individuals. The average uncertainty for the Lund (1996) major tie points was 2.8% vs. 5.1% for the Flathead dates. One of the Lund (1996) dates was particularly problematic (D11, uncertainty = 19.3%), contributing the bulk of the uncertainty for the major tie points. The higher uncertainty for the Flathead carbon dates is likely due to these dated stratigraphic points fortuitously being located on a sloped segment of the inclination or declination curve versus at an obvious maximum or minimum which likely would have made the tie points easier to consistently identify.

Interpolation Methods

All PSV age models use some interpolation method to construct the age model between a limited number of evenly or irregularly spaced dated points. However, no standardized interpolation function has been established, and the subject has received very little discussion in recent studies that have utilized PSV correlation dating. In fact, very few studies mention the interpolation method utilized, creating difficulties in comparing results and determining uncertainties (Lund 1996), even though changes in the interpolation function utilized (i.e., linear, polynomial, cubic) or parameters selected (i.e., 3rd or 4th degree) will yield very different overall age models.

The simplest age model would be a linear interpolation between points, which has the advantage of including each of the actual date points in the model (Hanna and Verosub 1988; Blais-Stevens et al. 2001; Verosub et al. 2001). However, a linear model method

requires the derivation of separate equations for each curve segment. This yields a fixed sedimentation rate for each segment and creates an unrealistic, step-function type model for sedimentation rate that can imply the presence of an unconformity at each step boundary. Interpolation with a polynomial fit (Verosub et al. 1986; Breckenridge et al. 2004) has the advantage of providing a single equation for the modeled chronology and yields a varying sedimentation rate that is smoothed to an extent that depends on the sampling interval. However, polynomial models may not pass directly through all the date points and tend to suffer from boundary effects at the top and bottom of the PSV data set, thus posing a different type of problem with these models.

In this study I examined linear and polynomial models to evaluate their effects on the final chronology and to establish the most realistic age model based on fit to the tie points and the geological reasonability of the computed sedimentation rate curve (see discussion below) for Flathead core 9P. I considered each interpolation model for declination and inclination data sets independently. For core 9P, I developed the age model to a depth of 622 cm. This approach includes the last interval of hemipelagic deposition, just above the uppermost upward-fining deposit.

I examined five polynomial models for core 9P, two of which (Full 3 and Full 4) modeled through the Mazama tephra with a date for the tephra located in the middle. Two other models (Gap 3 and Gap 4) compressed the depth scale for the Mazama tephra to a single centimeter. Once the Gap polynomial equation was developed it was applied to the entire 35 cm of the tephra to create the age model. In both the ‘full’ and ‘gap’ approaches, I

calculated 3rd and 4th degree polynomials. In the last approach to modeling the tephra, I split the data set to develop two polynomial equations, one above and one below the tephra. All the polynomial equations were developed using the core 9P tie points (Table 5) on Grapher v.5 software; models were calculated using an Excel spreadsheet. The results of the polynomial modeling are shown in Figure 25 and the calculated sedimentation rates are shown in Figure 26.

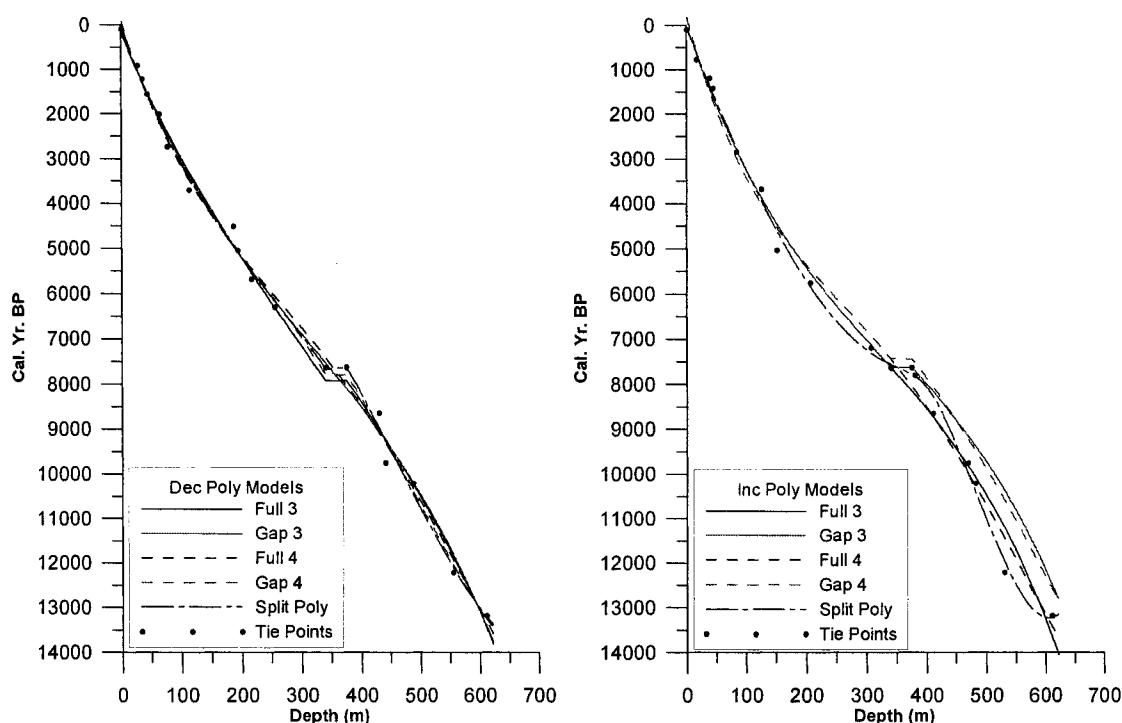


Figure 25 – Age models for declination (left) and inclination (right) are considered separately. Linear model represented by tie points, line not shown. Dates are based on average dates for each tie point calculated by Lund (1996) or tie points derived from Flathead cores (see Table 5).

As mentioned, polynomial interpolation functions are not required to pass directly through any date point and as such are subject to boundary effects at the top and/or

bottom of the data set. All polynomial models shown in this study suffered, to varying degrees, from this boundary condition. For example, the first centimeter of core 9P has a calculated $^{210}\text{Pb}/^{137}\text{Cs}$ date of 92 cal. yr. BP. However, the modeled polynomial dates ranged from -83 to 193 cal. yr. BP. These boundary effects were actually compounded when higher degree polynomials were attempted (not shown). To evaluate the best fit polynomial, I calculated a correlation R^2 between the tie point dates and the date modeled by each polynomial and the % Standard Deviation (%StDev) of the difference (Table 6).

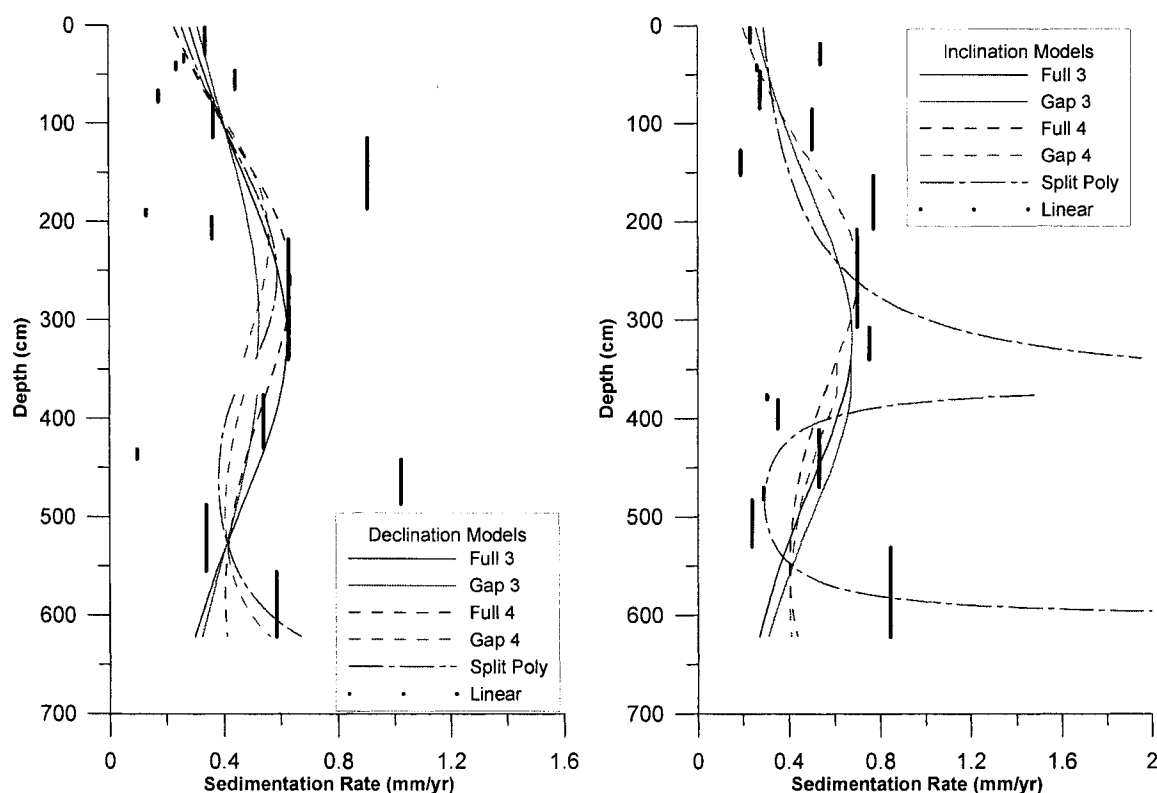


Figure 26 – Comparison of sedimentation rates for each of the considered age models. Declination (left) and inclination (right) are considered separately. Linear model shown as bars. Dates are based on average dates for each tie point calculated by Lund (1996) or tie points derived from Flathead cores (see Table 5).

I also calculated the %StDev without the first date, which suffered from the strongest boundary effects. The average uncertainty for the polynomial age modeling was 5.03% for declination and 11.35% for inclination at 1σ and is calculated as the average standard deviation of the percent difference of each modeled date to each tie point date.

Statistical measurements are only part of the model selection process. The selected model also has to be sedimentologically valid. Whereas this approach involves some subjectivity, I based the evaluation on a few key points: 1) we know the top of the core does not have a negative date, 2) the tephra dates for the core should be closely modeled, and 3) that the overall model fits well with all the ^{14}C dated points. Overall the split 3rd degree polynomial presented the best fit to the data. This model had an average difference of 9.02% to the tie points and an average $R^2=0.98704$. While these are not the best statistical fit, Paillard (1996) noted

“Unfortunately, the ‘fit’ is not always as good as with the simple visual correlation. A mathematical measure such as a correlation coefficient will indeed give more weight to the large timescale signal fluctuations (low-frequency variations) where much of the variance is located, than to the rapid ones that usually account for little of the variance.”

Model fits for the declination data have lower variability than the model fits for the inclination data (%StDev 5.03 vs. 11.35). For reasons not fully understood, the inclination models seem to be affected to a greater degree by the boundary conditions,

Model	Declination	Inclination
Polynomial Full 3	$R^2 = 0.995114$ %StDev = 4.95% %StDev w/o top = 3.78%	$R^2 = 0.991249$ %StDev = 12.08% %StDev w/o top = 6.11%
Polynomial Full 4	$R^2 = 0.995727$ %StDev = 43.59% %StDev w/o top = 3.36%	$R^2 = 0.992798$ %StDev = 71.14% %StDev w/o top = 8.52%
Split Polynomial	Upper $R^2 = 0.993476$ Lower $R^2 = 0.980604$ %StDev = 21.17% %StDev w/o top = 3.75%	Upper $R^2 = 0.994731$ Lower $R^2 = 0.988664$ %StDev = 7.00% %StDev w/o top = 5.70%
Polynomial Gap 3	$R^2 = 0.995606$ %StDev = 35.88% %StDev w/o top = 4.41%	$R^2 = 0.991249$ %StDev = 11.94% %StDev w/o top = 6.24%
Polynomial Gap 4	$R^2 = 0.99672$ %StDev = 27.86% %StDev w/o top = 3.81%	$R^2 = 0.992798$ %StDev = 70.73% %StDev w/o top = 7.90%
Table 6 – Statistical measures for polynomial age models for core 9P. All statistics are between the modeled dates and the tie point dates in Table 5.		

particularly at the bottom of the core. These effects are dramatically seen in the split inclination model's sedimentation rate (Fig. 26). This observation may be due, in part, to a down core compounding effect of inclination-shallowing, a systematic decrease in inclination angle as sediments are compressed (Verosub 1977; Rolph et al. 2004).

Reference Source Uncertainty

In PSV correlation dating the premise is simple: correlate a declination or inclination reference record to your data set and transfer over the dates to the appropriate part of the undated record. The general application of this method has been shown to be valid (Verosub 1988; Butler 1992; Rolph et al. 2004) and correlations can be made globally

(Itota et al. 1997). However, determining exactly what date to assign a particular tie point is more complicated. Logically the optimal situation would arise from correlation of dated points from a local well-dated reference lake in which magnetic drift and local magnetic anomalies are minimized (Lund and Banerjee 1985).

Although local well constrained PSV records are not always available, regional lacustrine reference records have been developed from several well-dated lakes (Turner and Thompson 1982; Lund 1996). And, standard tie points that can be identified across North America and globally have been established for the Holocene (Lund 1996; Itota et al. 1997) and are being developed for the Pleistocene (Steve Lund, written comm.). While these standard tie points can be correlated globally, each regional reference lake assigns slightly different dates to these points due to the cumulative effects of: 1) modeling differences, 2) dating uncertainty, 3) local magnetic anomalies, and 4) magnetic drift (which is poorly constrained). Thus, choices need to be made as to which reference data set to use.

All dating methods contain some amount of uncertainty, and radiometric dating is no exception. For direct ^{14}C dates, the instrument uncertainty typically is reported and, once converted to calibrated years BP, an uncertainty range based on the calibration curve is added to the instrument uncertainty. In the case of regional reference lakes the actual radiometric dates rarely fall on the curve minima and maxima that are most easily used in the correlation, requiring some interpolation to estimate dates between maxima and minima tie points.

The best correlations for core 9P would be derived from a radiometrically well-dated source from within the lake basin, but this source does not currently exist. Some locally derived PSV date points exist within Flathead Lake (i.e., FL-03-15K and FL-03-16K), but these dates are limited and are concentrated between the two tephras.

Geographically, Flathead Lake is located between Fish Lake (~500 km west) and Lake St. Croix (~800 km east); chronologic correlation with either of these reference records can be conducted for the Flathead Lake data set. Additionally, a set of average dates for the major tie points across North America have been calculated, providing another potential reference chronology and another source of uncertainty (Lund 1996).

In this study I examined the potential impacts and uncertainty of utilizing these three reference chronologies. Average dates for the major tie points were obtained from Lund (1996, Appendix B) and calibrated using OxCal 3.9 (Table 7). To compare the reference source differences, each of the correlated tie points were plotted (Fig. 27) using the tie point dates derived from Fish Lake, Lake St. Croix and the North American average dates (Table 7, Lund and Banerjee 1985; Verosub et al. 1986; Lund 1996). The tie point reference dates including the tephras dates were modeled using a 3rd degree polynomial (Fig. 27). Inclination and declination models using the average North American dates (Fig. 27A) graphically show a higher degree of similarity than the Fish or St. Croix chronologies (Fig. 27B & C). When visually comparing the inclination and declination chronologies from all three-reference sets (Fig. 28), it is apparent that the North American dates have a higher association to the St. Croix dates above the Mazama tephra and Fish Lake below the tephra. Uncertainty between the three chronologies as

	N. American Average Age ¹⁴ C Yr. BP	N. American Calibrated Age Cal. Yr. BP	Lake St. Croix ¹⁴ C Yr. BP	Lake St. Croix Cal. Yr. BP	Fish Lake ¹⁴ C Yrs. BP	Fish Lake Cal. Yrs. BP
Inclination						
1	870 ± 180	780 ± 185	960 ± 90	870 ± 190	1190 ± 90	1120 ± 170
2	1250 ± 180	1190 ± 185	1310 ± 140	1250 ± 300	1525 ± 105	1480 ± 220
3	1555 ± 180	1420 ± 185	1520 ± 40	1415 ± 105	1915 ± 65	1850 ± 150
6	2735 ± 380	2840 ± 400	2530 ± 130	2625 ± 325	2870 ± 90	3050 ± 300
7	3415 ± 250	3680 ± 275	3425 ± 145	3725 ± 375	3715 ± 145	4075 ± 475
9	4475 ± 240	5200 ± 245	4385 ± 135	5000 ± 450	4635 ± 105	5275 ± 325
FL-I1	4500 ± 250	5600 ± 250	--	--	4500 ± 250	5600 ± 250
10	6230 ± 300	7190 ± 325	6315 ± 95	7210 ± 220	6300 ± 130	7150 ± 350
12	6990 ± 300	7795 ± 325	6955 ± 65	7800 ± 140	6855 ± 155	7700 ± 300
14	8400 ± 470	9400 ± 750	9200 ± 200	9210 ± 190	8225 ± 115	10425 ± 275
Declination						
1	965 ± 180	915 ± 185	955 ± 75	875 ± 175	1045 ± 175	975 ± 325
2	1295 ± 270	1215 ± 325	1225 ± 55	1135 ± 145	1455 ± 85	1360 ± 180
4	1670 ± 60	1550 ± 95	1640 ± 80	1530 ± 190	1910 ± 60	1850 ± 150
5	2060 ± 180	2000 ± 245	2065 ± 105	2075 ± 275	2415 ± 85	2525 ± 225
6	2530 ± 300	2730 ± 425	2460 ± 140	2500 ± 350	2740 ± 100	2875 ± 375
7	3435 ± 320	3710 ± 450	3415 ± 135	3325 ± 375	3670 ± 100	4000 ± 350
9	4025 ± 270	4515 ± 275	4165 ± 145	4750 ± 600	4365 ± 65	5050 ± 230
10	4330 ± 500	4875 ± 700	4415 ± 105	5075 ± 275	4570 ± 40	5245 ± 205
11	4925 ± 380	5675 ± 600	5070 ± 120	5875 ± 325	5105 ± 105	5900 ± 300
12	5485 ± 360	6295 ± 450	5560 ± 130	6325 ± 375	--	--
15	8060 ± 360	8975 ± 450	8140 ± 200	9100 ± 350	7855 ± 125	8730 ± 250
16	8280 ± 350	9100 ± 500	8705 ± 165	9825 ± 325	8095 ± 45	9115 ± 125
17	8375 ± 320	9300 ± 300	9130 ± 130	10340 ± 160	8250 ± 90	9220 ± 190

Table 7 – Tie point data, except FL-I1, taken from (Lund 1996) showing date variation from well dated lakes. North American data shown includes dates from lakes not used in this secular variation study, but are included to constrain dating uncertainty. FL-I1 calculated based on a Fish Lake curve date. Data in table only includes tie points identified in reference lakes and not all were identified in core 9P. Radiometric calibration using 1-σ error values in cal. yr. BP from OxCal 3.9.

average percent standard deviation of the differences at 1σ is 7.07% for declination and 11.3% for inclination. While the date uncertainties for the reference lakes themselves are lower than the average North American dates, the uncertainties for the reference lakes are only stated as the radiometric uncertainty and do not include any modeling uncertainty (Fig. 28).

Longitudinally based variation in paleomagnetic secular variation is well documented but poorly constrained and not well understood (Verosub et al. 1986). The PSV drift affects the timing of locally derived minima and maxima on the PSV data curves (Lund 1996).

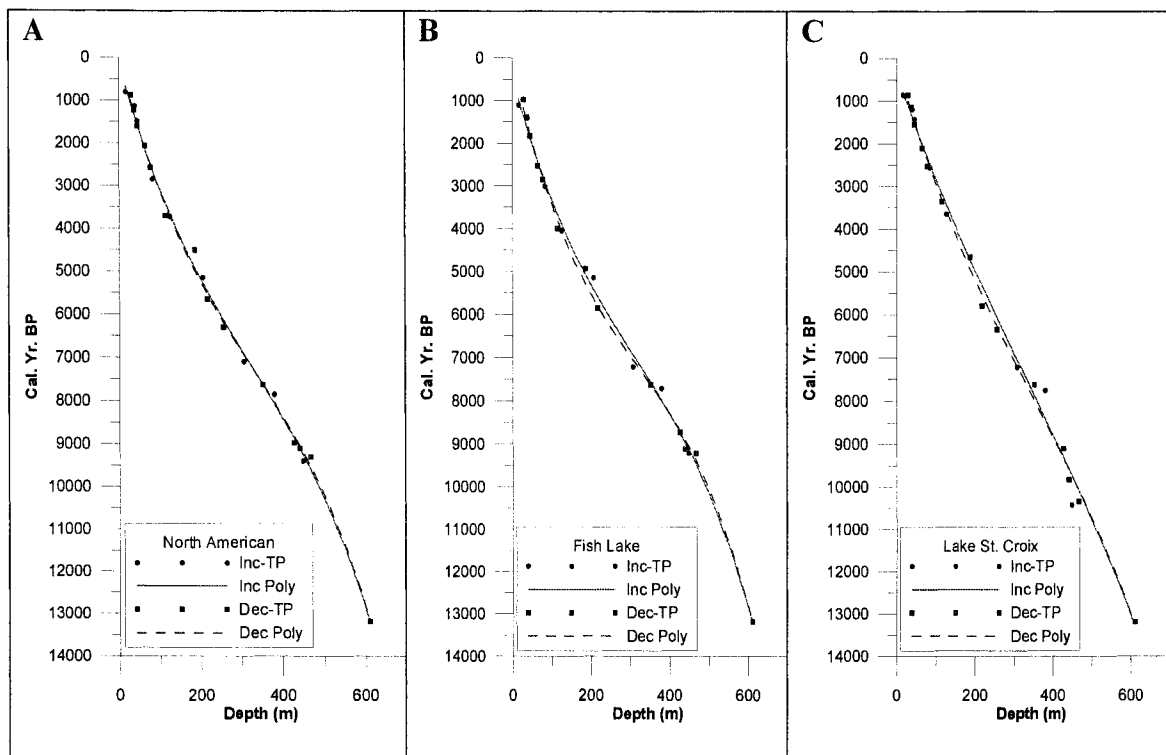


Figure 27 – Comparison of date models for core 9P based on tie points identified by Lund (1996) and the Flathead tephras, using calculated average dates and dates from reference lake (Fish Lake and St. Croix Lake) age models. All dates from Lund (1996). Notes: TP=tie points, Poly=Polynomial, Dec=declination, and Inc=inclination

Minimizing date variability can be achieved by selecting, when available, a reference lake close to the record of interest (Verosub et al. 1986), thus reducing local magnetic disturbances and PSV drift problems. However, with the presence of these types of discrepancies, utilizing an average date model spread over a number of localities seems advantageous by assigning more realistic dates despite the higher average uncertainties (Lund 1996). Uncertainty for the average North American dates is large enough to encompass the local variation in the Fish and St. Croix polynomials at most of the tie

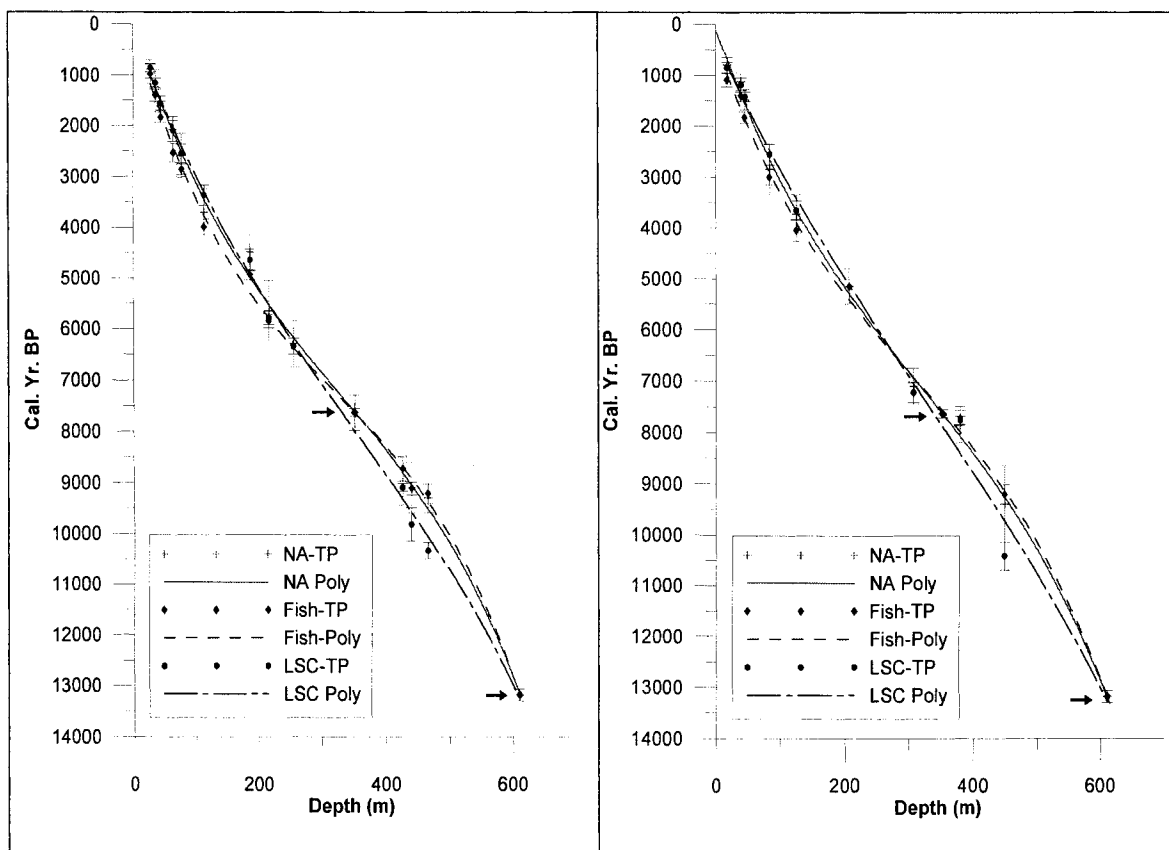


Figure 28 – Comparison of tie points dates for three possible chronologic models. Plots for core 9P declination curve on left; inclination curve on right. Tie point dates shown and associated 3rd degree polynomials based on reference lake date data and North American average dates (Lund 1996, Appendix B). Uncertainty bars shown for each date point. Tephra noted with arrows, in the declination graph a tie point overlies the Mazama tephra.

points (Fig. 28). The North American calibrated uncertainty ranges from ± 80 to ± 700 yrs for the declination data and from ± 185 to ± 750 yrs for the inclination, with an average uncertainty of ± 353 yrs and ± 313 yrs respectively. These average North American dates for the tie points represent the most conservative reference set for the establishment of a the chronologic model.

Final Model Considerations

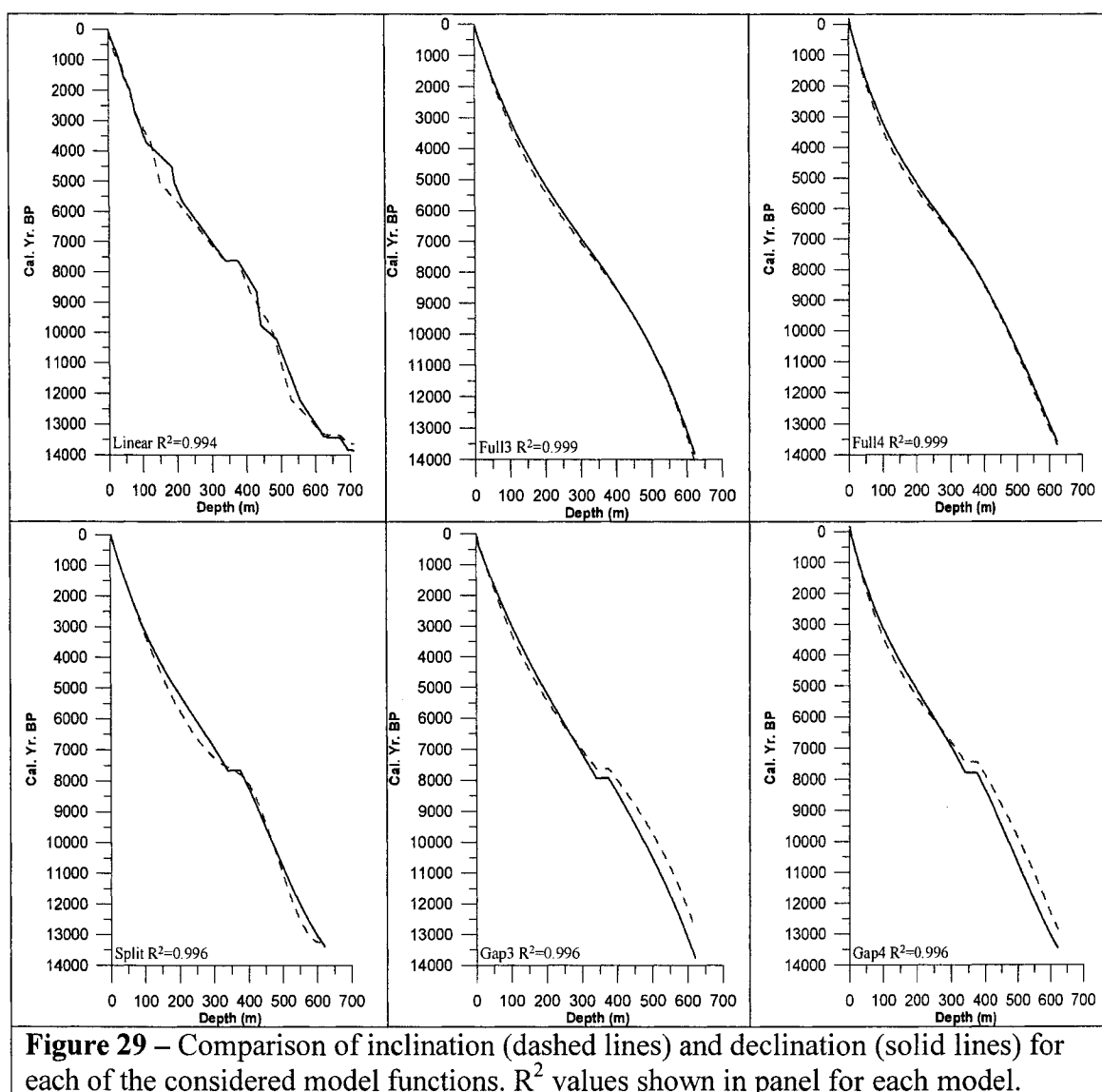
In constructing a correlative chronostratigraphy based on PSV data sets, it is the researcher's job to interpret the data and present the most parsimonious model with the lowest acceptable uncertainty. It is not unusual for the researcher to select sections of various records, with justification, to compile the final model (Gary Acton 2004, pers. comm.). Construction of a composite chronologic model in this way allows the best fit solution to be used and can constrain uncertainty (Talling and Burbank 1993). Care must be exercised to assure all chronologic data sets use the same absolute dating schemes (e.g., radiocarbon years before present, ^{14}C BP, vs. radiocarbon calendar year, ^{14}C Cal., vs. calibrated years before present, cal. yr BP). Many of the data sets utilized in this research were published as ^{14}C BP dates. I converted these radiocarbon years to calibrated years, using OxCal calibration software version 3.9 developed by University of Oxford's Radiocarbon Accelerator Unit (<http://www.rlaha.ox.ac.uk/orau/oxcal.html>). All presented figures in this study use cal. yr. BP.

In constructing the final age model for core FL-00-9P I started with the two tephras and correlation of $^{210}\text{Pb}/^{137}\text{Cs}$ data from core 9G to constrain the top of the core. For further

refinement of the core 9P chronology, I investigated the potential PSV correlation with well-dated reference lakes. From these investigations I conclude that PSV is an effective method for dating a core where limited datable material is available. However, the uncertainty of the method is greater than the few hundred years claimed by Hanna and Verosub (1988). When considering uncertainties from tie point selection, modeling, and reference sources, I estimate average uncertainty at 300 to 400 years for dates that are derived from outside the local basin. This uncertainty is reflected in the average North American dates that were calculated by Lund (1996) and that were selected for use in the conservative approach I adopted in constructing the chronology for core 9P. In addition to the PSV reference dates published by Lund (1996), I also used dates available from other Flathead Lake cores. These intrabasinal dates likely do not suffer from longitudinal variation, model extrapolation, or regional magnetic anomalies to the extent that the average reference dates published by Lund (1996) do and thus reduce uncertainty, where utilized.

The overall chronologies based on declination and inclination tie points have a high degree of correlation, $R^2 =$ of 0.994 for a linear model. The fit between declination and inclination can be improved based on the tie point model selected (Fig. 29). For example, a simple regression of the 4th degree polynomial model has a $R^2 =$ of 0.999, while Split model has an $R^2=0.996$. However, differences are apparent from the uncertainties among the declination (5.03%) and inclination (11.35%) models (Table 6). Based on the lower uncertainty I elected to model core 9P utilizing the declination tie points. To model the final chronology for core 9P, I selected a fit function using the split 3rd degree polynomial

as previously discussed (Figs. 25, 26 & 29). This split declination model has an average percent difference of 9.02% from the tie points and the best overall fit to the date points contained in core 9P. However, this modeling function may not be the best fit for all data sets due to the repeated tephra section and I would suggest modeling each core individually. Lastly, for the upward-fining interval below the Glacier Peak tephra I



utilized the model developed in the initial chronology that multiplied the percent of hemipelagic deposition of each centimeter by the sedimentation rate modeled by the polynomial. To compensate for the boundary effect on the sedimentation rate at the bottom of the core, I used the average sedimentation rate (0.51 mm/yr) calculated between the last two tie points (555 and 611 cm).

Chronostratigraphic Model and Uncertainty

The final chronostratigraphic model for FL-00-9P (Fig. 30 & 31) was constructed based on the considerations discussed above. The goal was to develop a model with the greatest sedimentologic authenticity and the least uncertainty. To this end, the initial model has been refined with PSV correlations and the transfer of the tie point dates to core 9P. Above the Mt. Mazama tephra the nine declination tie points were derived from a PSV Holocene study by Lund (1996), one correlation to core 16K, and the $^{210}\text{Pb}/^{137}\text{Cs}$ correlation to core 9G (Fig. 30). Uncertainty in this portion of the core was calculated as the date point uncertainty plus 7.6% for polynomial modeling uncertainty for the top portion of the core, plus 2.8% for the point selection uncertainty. Between the tephras, four declination tie points were correlated to core 9P from Flathead cores 15K and 16K (Fig. 30). Uncertainty of these tie points was calculated as the date uncertainty plus 1.05% for the modeling uncertainty, plus 5.1% for the point selection uncertainty. The uncertainty for the tephra radiometric dates is calculated as the date uncertainty plus the modeling uncertainty of 1.05%.

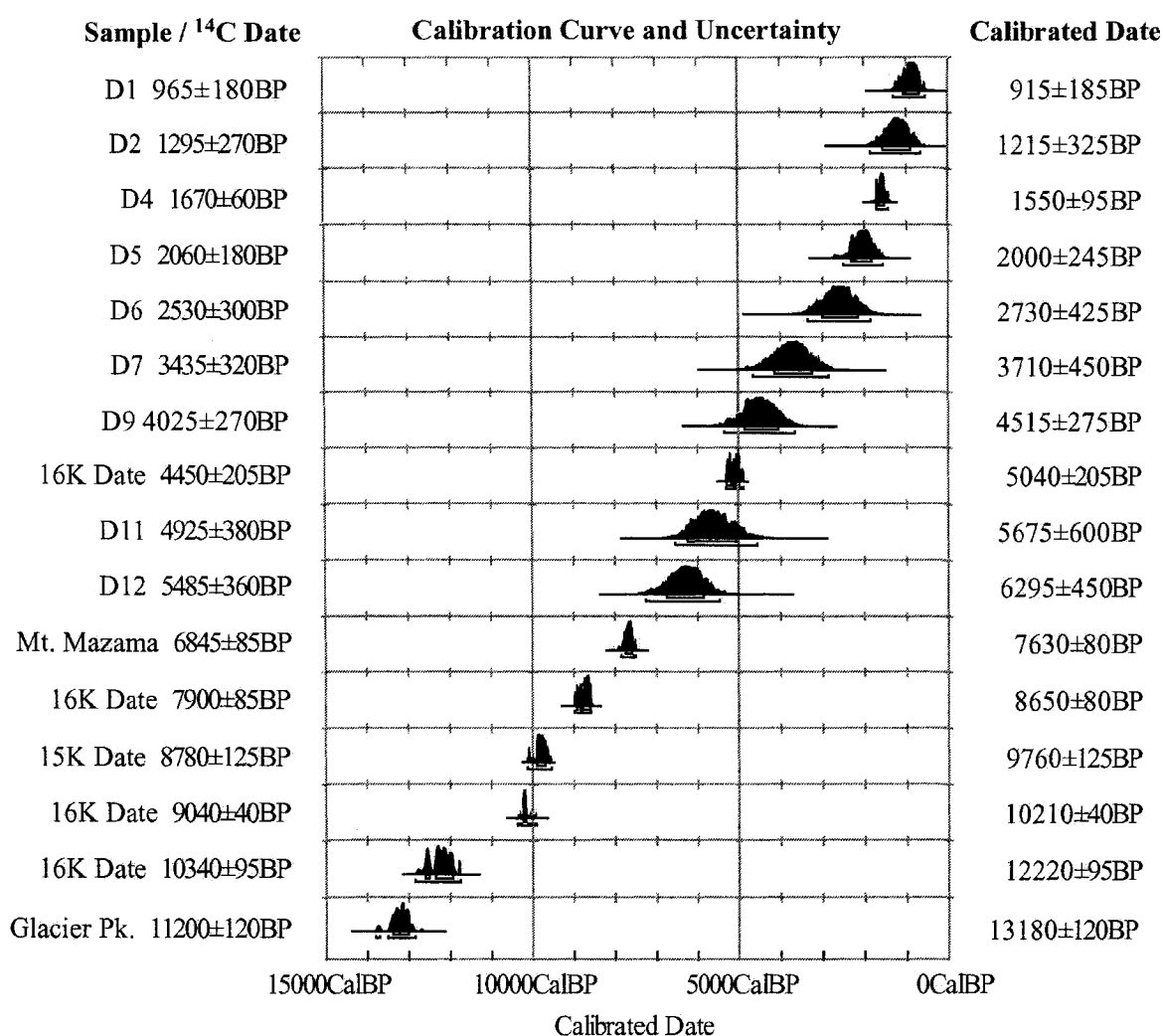


Figure 30 – Tie points for the final chronostratigraphic model for core FL-00-9P. Tie point and ^{14}C BP date shown on left. Graphs and calibrated years BP (right) calculated on OxCal.

The refinement of the age model with PSV correlations did strengthen the chronology by providing an expanded data set that confirmed the initial simplistic model and allowed a more realistic depositional model to be created (Fig. 31). The refined model above the Mazama tephra differs slightly from the simple model but is generally within the uncertainty. However, the uncertainty in this portion of the model is greater due to the

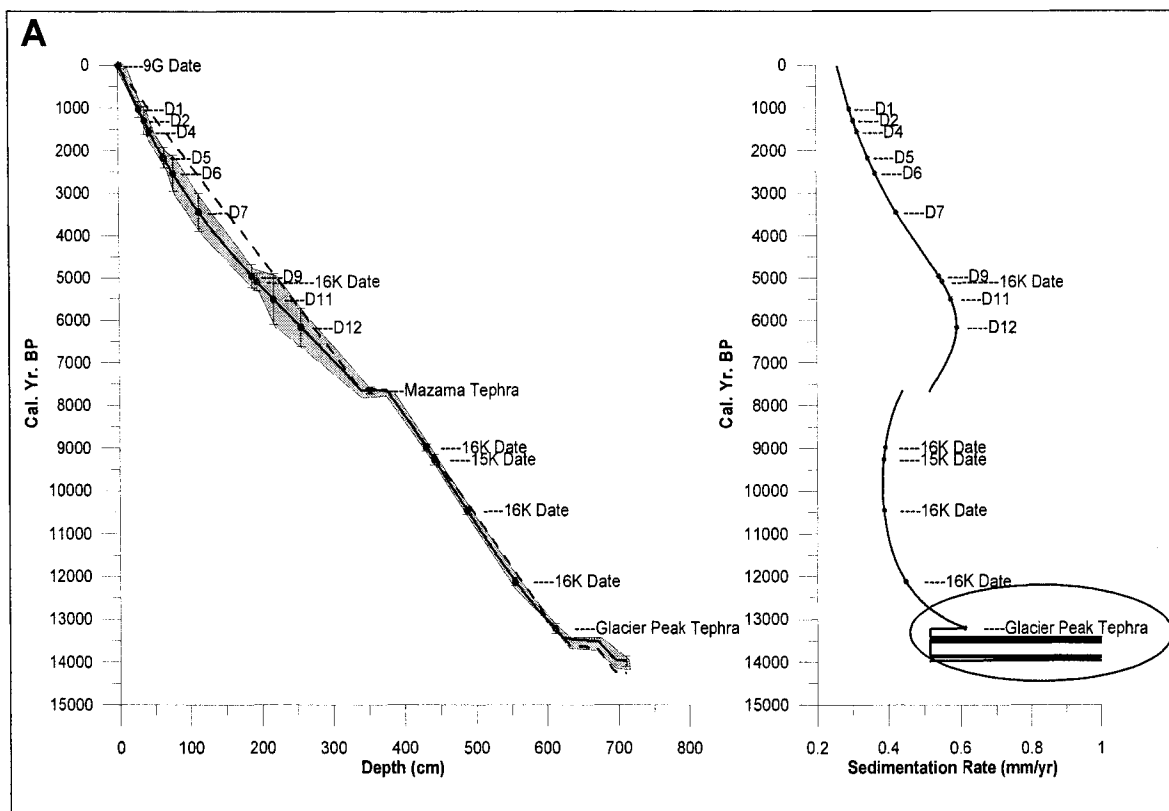
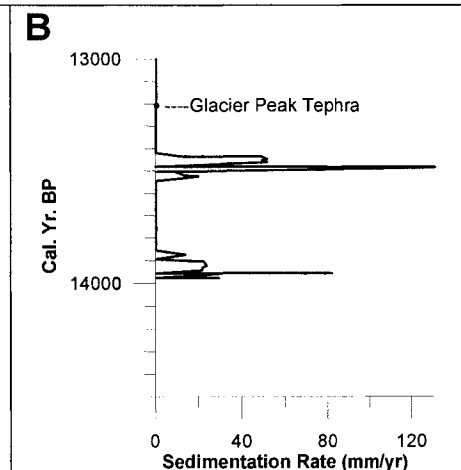


Figure 31 – (A) Final chronostratigraphic model for core FL-00-9P. Age model plotted for time vs. depth, points of correlation are shown with dots. Uncertainty shown with error bars for each tie point and with gray shadow range. Dashed line is initial age model. Graph on right shows sedimentation rate (unadjusted for water content) based on modeled timescale, bottom peaks truncated at right end of x-axis (oval). (B) Truncated portion of sedimentation rate graph (oval in A) showing sedimentation rate associated with upward-fining beds drafted to scale.



limited dateable material in the Flathead basin. When utilizing tie point dates from within the basin between the tephras, the refined age model is nearly identical to the simple model and uncertainty is greatly reduced (Fig. 31). The overall agreement of the models strengthens the suggestion that the core 9P does not contain major gaps or rotations and it can provide a complete paleoclimatic record for the Holocene and late

Pleistocene. Additionally, this study has demonstrated that PSV correlations can be quantified and used to provide a basis for age modeling within the uncertainty limits discussed.

Sedimentologic Model Validation

One of the criteria to consider when constructing a chronological model is the sedimentologic validity of the input parameters and the model created. In this study independent data sets can be utilized to test the soundness of the chronologic and sedimentation rate models. As previously stated no known unconformities exist in core 9P, with the exception of possible missing record due to Mazama tephra slumping. The step-function style sedimentation rate of the linear model implies that unconformities exist where no other data support this suggestion. The selected split model used in this study does have independent support in that the declining sedimentation rate between ~12,000 and 13,000 cal. yr. BP, corresponds to a period of glacial retreat (Smith 2004; Hofmann 2005) and lower hydrologic flow (lower median grain size). A shift in the sedimentation rate at the Mt. Mazama tephra accounts for the erosional unconformity due the tephra slumping. And, widespread seismic onlap onto the Mazama reflector (Hofmann 2005) indicates a lake level lowstand immediately after deposition of the Mazama tephra, followed by an increase in hydrologic flow during the lake refilling. The chronologic model for core 9P shows acceleration in the sedimentation rate beginning above the Mazama tephra (Fig. 31). These independent sedimentologic supports provide additional strength to the discussed modeling decisions used to construct the core 9P chronology.

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CHAPTER 3

Examination of Potential Paleoclimate Proxies for Large Open Lacustrine Systems: Late Pleistocene and Holocene Time-Series Core Data from Flathead Lake, Montana

Abstract:

Time-series proxy data acquired from lake sediments are routinely used to reconstruct regional climate histories. However, no standard set of proxies can be consistently applied to understand past paleoclimate, and significant complications can arise both from the integration of proxy signals over the area of the catchment basin and from processes of destructively interfering proxy signals within the watershed.

In this study I present the initial analysis and evaluation of a suite of proxies commonly utilized in paleoclimate reconstructions as derived from sediments in Flathead Lake. The investigations presented herein concentrate on sediment core FL-00-9P, recovered from the lake bottom in 2000. This study focuses on changes in grain size, carbon and nitrogen, and mineralogical data at a millennial to centennial time-scale. I present the methods utilized in obtaining these time-series data sets, critically review the use of similar proxies at other localities, and evaluate the overall effectiveness of each proxy as a tool for reconstructing paleoclimate. My results suggest that many proxies routinely used to reconstruct paleoclimate do not provide a statistically robust means of quantifying paleoclimatic variability in the Flathead Lake basin. This result may be partly due to a combination of factors, including periodic interference in the proxy signals from different climatic environs in the mountainous upper Flathead catchment basin, low signal/noise ratios within the datasets due to analytic uncertainty, and smoothing effects created by sediment recycling within the catchment.

Introduction:

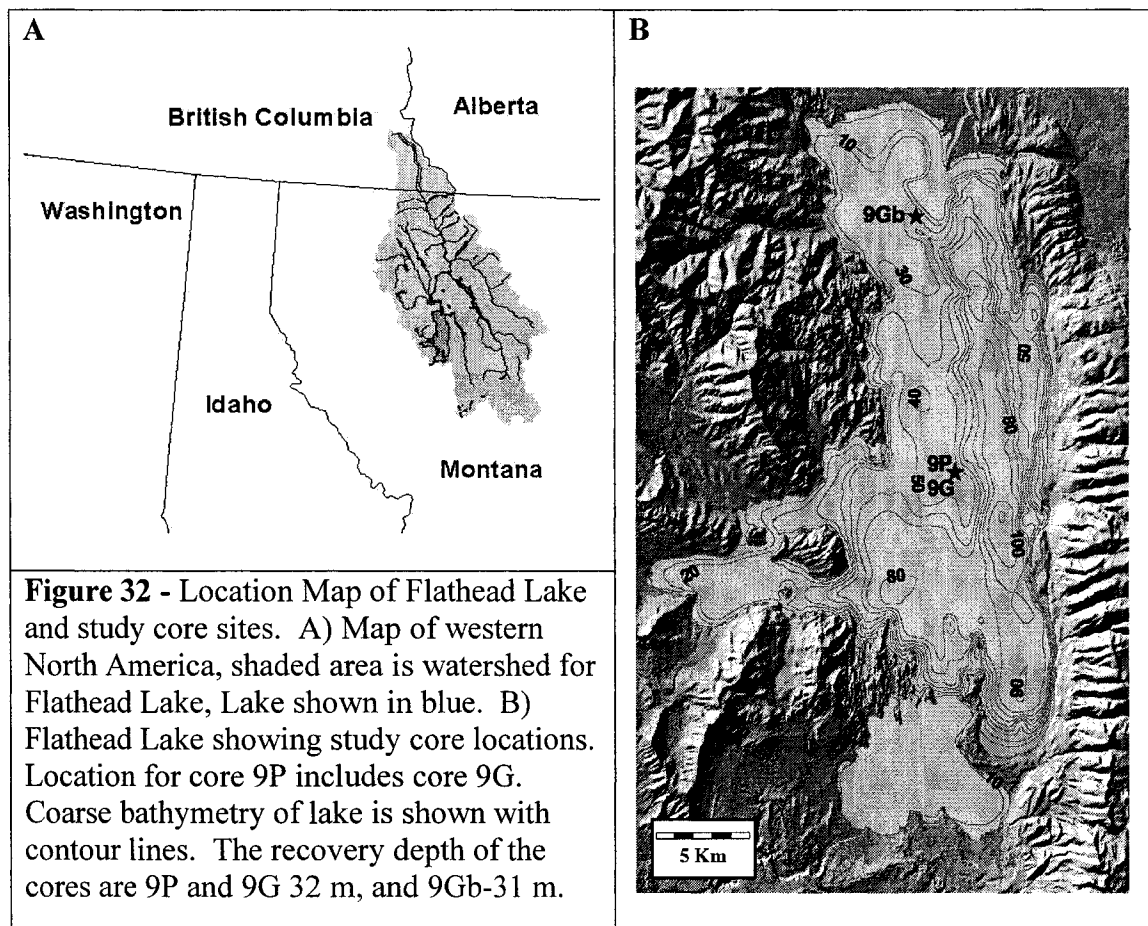
The record of past climatic variability is a powerful guide in understanding and predicting future natural climate change. In particular, understanding the range of prehistoric natural climate variability is important for placing ongoing anthropogenic modifications of climate into a longer-term perspective (Magny 1993; Alexander and Windom 1999; Einsele et al. 2001). In many published studies, the interpretation of ancient climates is achieved by the development of time-series data sets, called proxies, for variable parameters within natural records. A proxy data set is utilized as a substitute for one or more climatic, environmental, or physical conditions that existed in the past but cannot be measured directly (Henderson 2002). Proxies are presumed to vary predictably with changing climate and so represent a record of climate change. The proxies of ancient global and local climates can be preserved in a variety of natural records, including glacial ice sheets, ocean and lake sediments, tree rings, and corals (Patterson et al. 1977; Allen and Anderson 2000; Horiuchi et al. 2000; Sarkar et al. 2000; Wagner et al. 2000). Commonly used proxies for climate studies include variations in micro and macro fossils, isotopic ratios, magnetic susceptibility, sediment grain size, mineralogy, elemental composition, and carbon chemistry (Mathewes and Rouse 1975; Dean and Megard 1993; Forester and Carter 1998; Cohn 2003).

Lake sediment records have long been exploited as a source of paleoclimate proxy data. Lakes respond quickly to environmental change and may be characterized by long periods of uninterrupted sedimentation. Lacustrine climatic studies traditionally have concentrated on small closed lake systems, which are sensitive to local climate change

and allow researchers to work with a localized set of proxy input variables (Whitlock et al. 1993; Meyer et al. 1995; Long et al. 1998; Millspaugh et al. 2000). This approach may accurately record regional paleoclimate in some situations, such as the North American Great Plains where mountains do not increase microclimate variability (Szeicz and MacDonald 2001). However, in mountainous regions, closed lacustrine systems may also record local orographical microclimate effects that may not accurately reflect the greater regional trends. Local microclimatic variability can impact both temperature and precipitation, both of which are major components of a fluvial/lacustrine system (Szeicz and MacDonald 2001; Wang et al. 2002). Although the processes contributing to climatic variability between montane drainages are not completely understood, large open lake systems should integrate the variability of montane microclimates and record a more regional signature of climatic fluctuations derived from its watershed.

The main objective of this study is to construct and analyze a series of commonly used time-series proxy data sets from Flathead Lake, Montana, to investigate the utility of these proxies as recorders of ancient climate. In particular, I seek to conduct a rigorous analysis of analytic uncertainty to quantify the signal/noise ratio and test the hypothesis that these proxy data do record statistically significant variations that can be attributed to ancient climate change. This work seeks to test the hypothesis that, using time-series proxy data sets, a predictable record of paleoclimate change can be extracted from sediments in a large, open lake. In addition, I seek to test the hypothesis that microclimate variability in mountainous terrain integrates paleoclimate signals derived from proxy studies downstream.

The Flathead watershed (over 18,000 km², Fig. 32A) drains a network of mountainous and high valley terrain, and experiences variable climatic conditions due to the spatial heterogeneity of montane microclimates (Bartlein et al. 1998b; Rial and Anaclerio 2000; Weber 2001). Flathead Lake is the largest freshwater lake (496 km²) west of the



Mississippi with a maximum length of 43 km and width of 23 km. The lake is located at the former southern terminus of the Cordilleran ice sheet during the last glacial maximum (Fig. 33, Atwater 1986) between the Mission and Flathead Valleys in northwestern Montana. The Mission and Flathead Valleys are located at the southern end of a large

linear valley system called the Rocky Mountain Trench, that extends for approximately 1,600 km from northern Montana to the British Columbia-Yukon border (Leech 1966). The Mission and Flathead valleys are structural features created by the Mission Fault, a large down to the west normal fault, and is bounded on its eastern margin by the Mission Mountains (Fig. 34). Seismic reflection data of the fault beneath the lake however

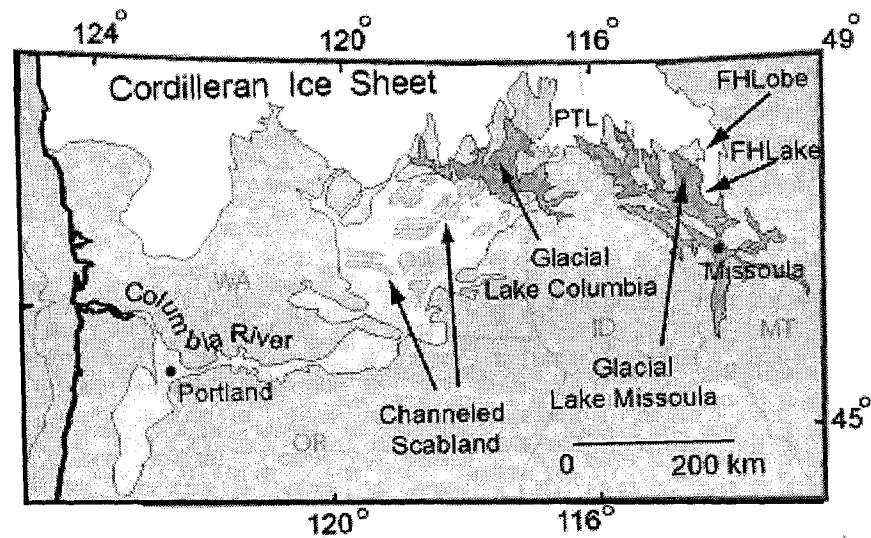


Figure 33 - Map of approximate maximum extent of the Cordilleran Ice Sheet in Western North America. Flathead Lake is positioned under the Flathead ice lobe (Hofmann 2005).

shows that the Mission Fault is a complicated system of extensional and strike-slip structures (Hofmann et al. 2006). Flathead Lake occupies a structural low in the valley that contained the Flathead Lobe of the Cordilleran ice sheet during much of the late Pleistocene (Fig. 33). Sedimentologic and seismic reflection data show that the Flathead Lake basin existed as a proglacial lake in the late Pleistocene and provides a record of glacial retreat and subsequent lake development over the transition to the Holocene

(Curry et al. 1977; Smith 1977; Chambers et al. 1989; Ostenaa et al. 1990; Levish 1997; Smith 2004; Bondurant 2005; Hofmann 2005; Timmerman 2005).

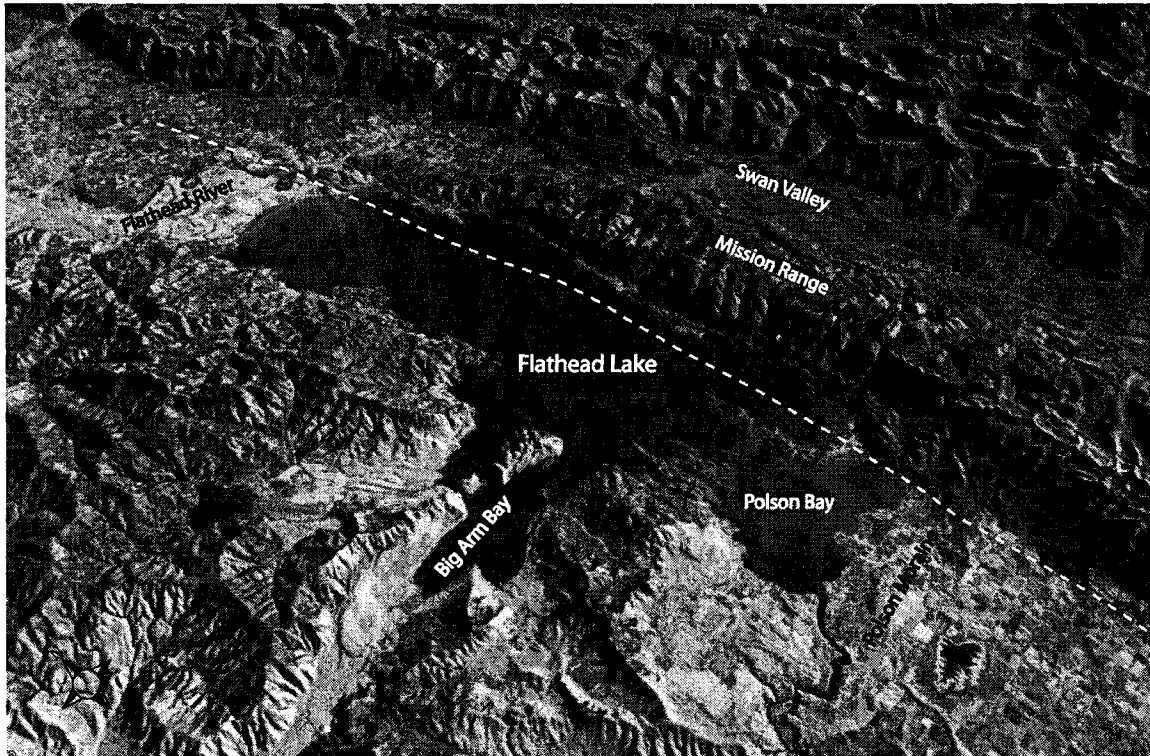


Figure 34 - Aerial image of Flathead Lake and the surrounding landscape. A major normal fault system runs through the eastern part of the lake forming the scarp face seen on the western side of the Mission Mountains (dash line approx. main fault location). Map image courtesy of William Bowen at the California Geographical Survey.

Flathead Lake currently has a mean surface elevation of ~881 m above sea level. Since the 1938 completion of Kerr Dam the lake level has been maintained at ~2 m higher than the mean annual elevation of 879 m prior to the dam construction. Current lake surface elevations range from 879 to 882 m versus the 878 to 881 m pre-dam range (Fig 35). On the western side of the lake a broad bathymetric bench exists with an average water depth of 35 m (Fig. 32B). The eastern side of the lake has a north-south trending bathymetric trough (Fig. 32B) that has a maximum depth of 117 m (Moore et al. 1982). Contained

within the sediments of the bathymetric trough is the sub-surface expression of the Mission Fault (Hofmann et al. 2006).

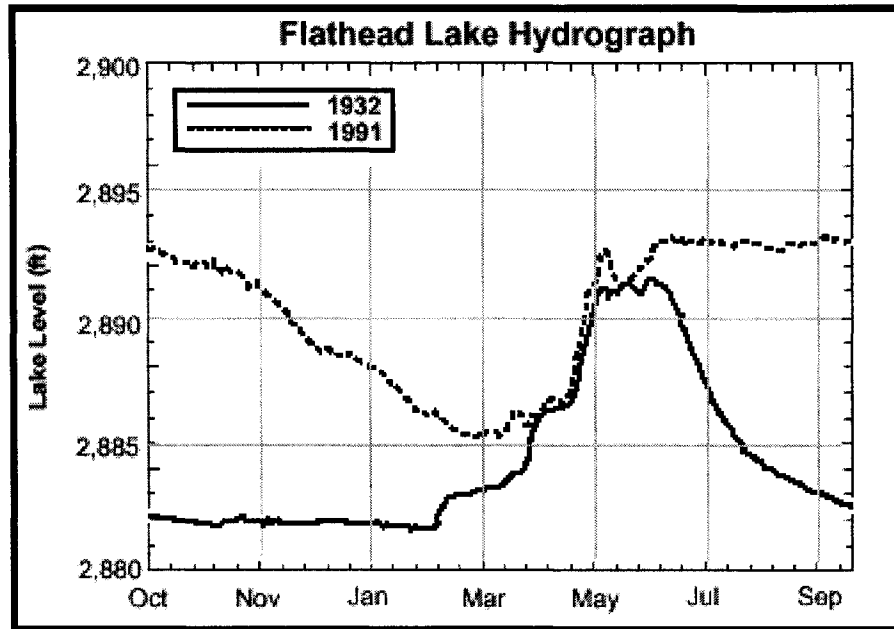


Figure 35 - Hydrograph of Flathead Lake showing spring runoff before construction of Kerr Dam (solid line) and post-dam (dashed line). Since operation of Kerr Dam began the Flathead Lake water level has remained high for most of the year to supply power generation and enhance recreation. The result has been transformation and erosion of the Flathead River delta front (Moore et al. 1982).

Flathead Lake is an open system that drains over 18,000 km² of the northern US Rocky Mountains and southern Canadian Rocky Mountains (Fig. 32). Based on stream discharge data from the USGS (<http://waterdata.usgs.gov/mt/nwis/dv?>) for the last 20-year period, approximately 86% of the lake's modern inflow is delivered by the Flathead River at the northern lake margin while an additional 10% is transported from the southeast part of the watershed through the Swan River. Annual discharge of the Flathead River is dominated by melting of the winter snowpack in the high altitude

regions of the catchment. Flathead Lake is oligotrophic, with relatively low rates of primary productivity (Moore et al. 1982; Moore 1983) due to limited nutrient loading (Spencer and Ellis 1990; Spencer and Owen 2004).

Methods and Results:

In 2000 Dr. Marc Hendrix and Dr. Johnnie Moore from the University of Montana Department of Geology undertook a new round of research focused on Flathead Lake and the surrounding basin. This new effort followed initial work on the basin and lake by Dr. Moore and others over 20 years earlier (i.e., Decker 1966; LaPoint 1973; Joyce 1980; Kogan 1980; Moore et al. 1982; Moore 1983). The initial focus of this new research was the documentation of climatic and hydrologic change during the late Pleistocene and Holocene (~16,000 cal. yr. B.P. to present) from analysis of sediment cores collected in the Flathead Lake basin. Material for these analyses was obtained in the fall of 2000 with the recovery of 8 piston cores and 6 gravity cores. Subsequently additional cores were recovered from Flathead Lake in the summer of 2001 (4 gravity cores and 2 freeze cores), the fall of 2002 (15 gravity cores), and the summer of 2003 (11 piston cores and 8 gravity cores). All the piston cores were recovered using the modified Kuhlenberg coring facilities from the Limnological Research Center at the University of Minnesota (Fig. 36). To maintain targeting control on subsurface structures during coring, the longer piston cores were mostly located along one of the 3.5 kHz seismic reflection lines recovered in 1979 (Kogan 1980).

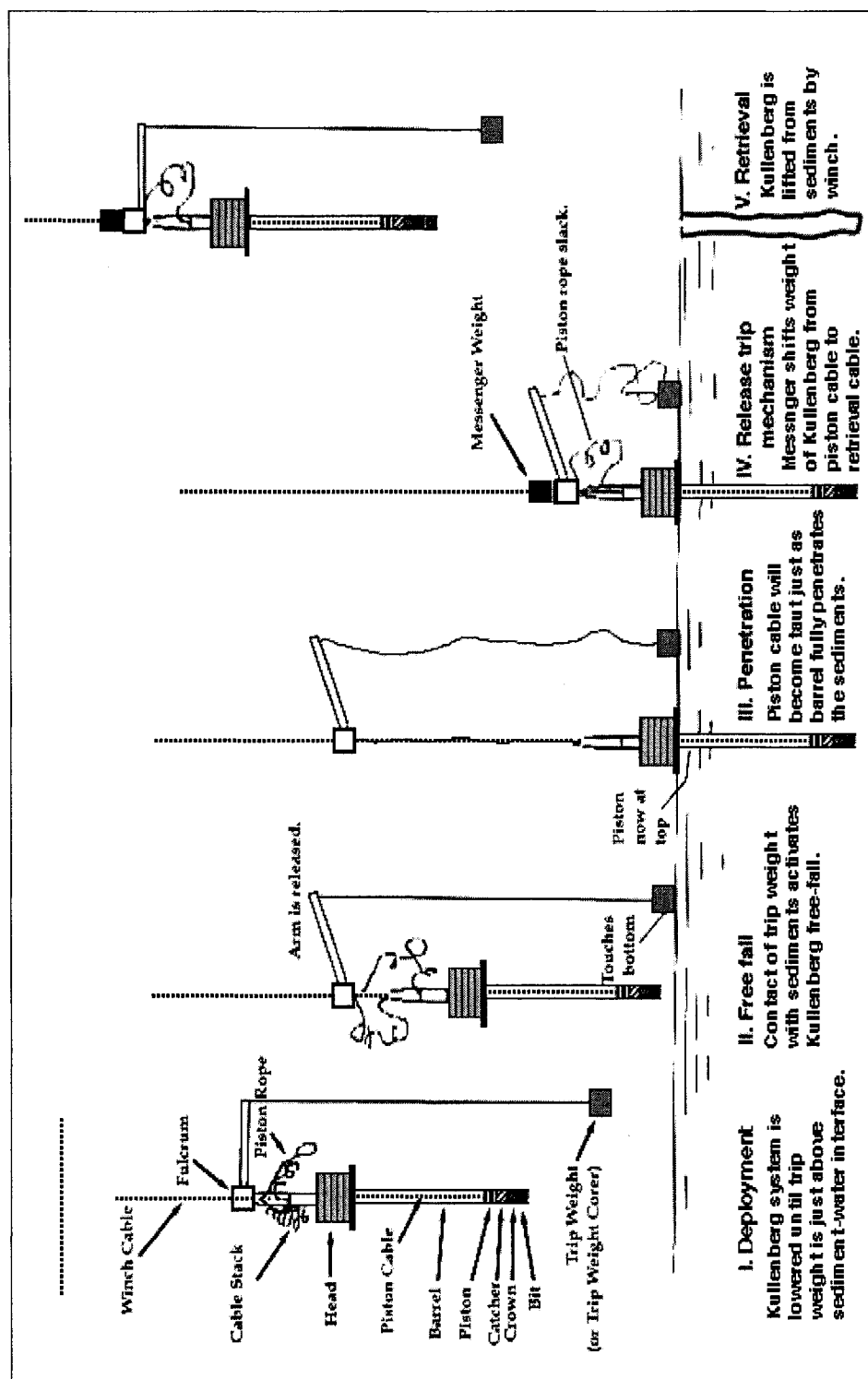


Figure 36 - Schematic of modified Kullenberg coring system (Graphic courtesy of Limnological Research Center)

General Core Processing:

The focus of this research is on core FL-00-9P (9P) that was recovered in September 2000 at a depth of 32 m, using the modified Kuhlenberg piston-coring platform (Fig. 36). The core location is in the center of the lake (UTM, N5308023.793 and E716377.5239), along seismic line 28 (Fig 37 & 38), one of the 3.5 kHz reflection lines recovered in 1979 (Kogan 1980). The core is situated atop a broad bathymetric bench that occupies the western portion of the lake (Figs. 32B, 37 & 38).

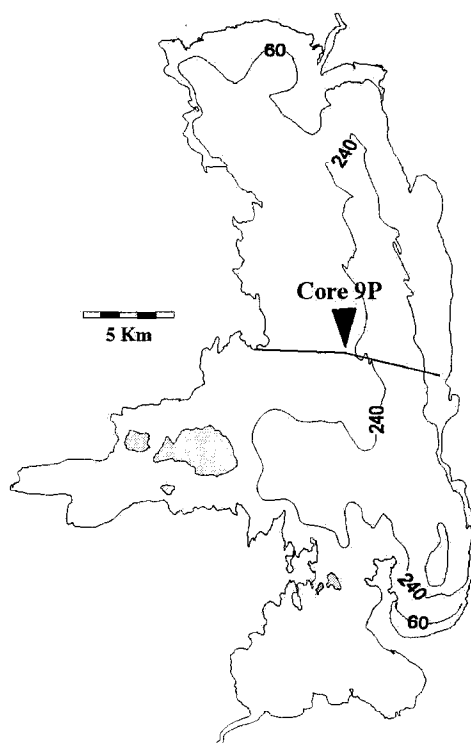


Figure 37. – Map of Flathead Lake showing position of core 9P on seismic line 28. Seismic data collected in 1979 (Kogan 1980) and core 9P recovered in 2000.

The recovered core includes only the top ~10% of the sediment package, based on the 3.5 kHz seismic reflection data that suggest that an intact sediment package over 60 m thick exists at the coring site (Kogan 1980; Hofmann et al. 2006). However, lower frequency seismic reflection data, which is deeper penetrating but of lower resolution, suggests an even thicker sediment package with a depth to bedrock of ~130 m (Wold 1982). Initial observations of core 9P suggest that it contains a continuous sedimentation record without any bioturbation.

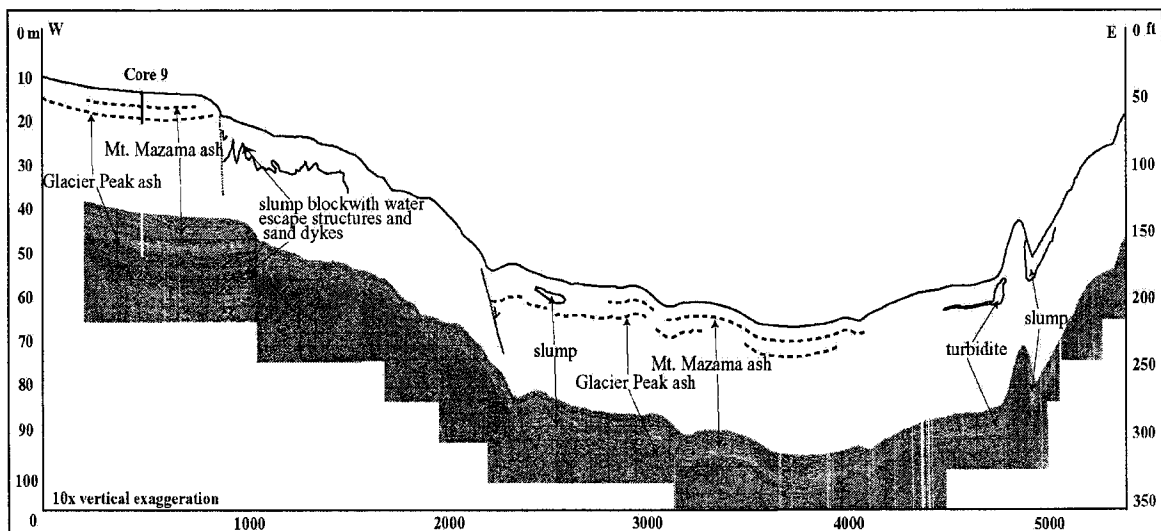


Figure 38 - Interpretation of seismic line 28 shows intact sediment package below Core 9P. The two dashed lines mark the position of the Mount Mazama (7,630 cal. yr. BP) and the Glacier Peak tephra (13,180 cal. yr. BP). The solid black line is the sediment-water interface. A strand of the Mission Fault can be traced on the west side of the trough; no fault is imaged on the east side of the trough in this seismic line. Slump structures and turbidites (possible seismites) appear to be between 15,000 years and 7,000 years old. (Modified from Hofmann and Hendrix 2002)

Core 9P is chronostratigraphically constrained by tephras from Mt. Mazama ($7,630 \pm 80$ cal. yr. BP) and Glacier Peak (GPA; $13,180 \pm 120$ cal. yr. BP). The tephras are located at 375 cm and 611 cm, respectively, below the lake floor and demonstrate that the sedimentary record extends to the Late Pleistocene. In this study all dates utilized are based on a chronology prepared for core 9P (Fig. 39) that was derived from the tephra dates, ^{137}Cs and ^{210}Pb isotope correlations, and paleomagnetic secular variation correlations (Chapter 2, this dissertation). In addition to core 9P, this study examines data from two gravity cores FL-00-9G and FL-02-9Gb. Chris Fuller of the USGS in Menlo Park, CA dated these gravity cores with ^{137}Cs and ^{210}Pb isotope methods.

In November 2000, core FL-00-9P was split along its long axis and photographed with a Leaf Microlumina digital camera equipped with a Nikon AF Micro Nikkor 60 mm lens. The images were processed and joined using Adobe Photoshop version 5.5 (Fig. 40). One half of the core was archived for future research, while the other half was sectioned at one-centimeter intervals (pucks) over its entire 7.10 m length for analyses. In December 2000, the archived half of the core was brought to the Curry Heath Center at The University of Montana to acquire X-radiograph images. The X-radiographs were processed and joined using Adobe Photoshop version 5.5 (Fig. 41). In August of 2002, the archived half of core FL-00-9P was transported, with the other piston cores, to the Limnological Research Center at the University of Minnesota for analysis using a Geotek Multi-Sensor Core Logger (Geotek) for physical property description. The Geotek collects data for magnetic susceptibility, P-wave amplitude and velocity, impedance,

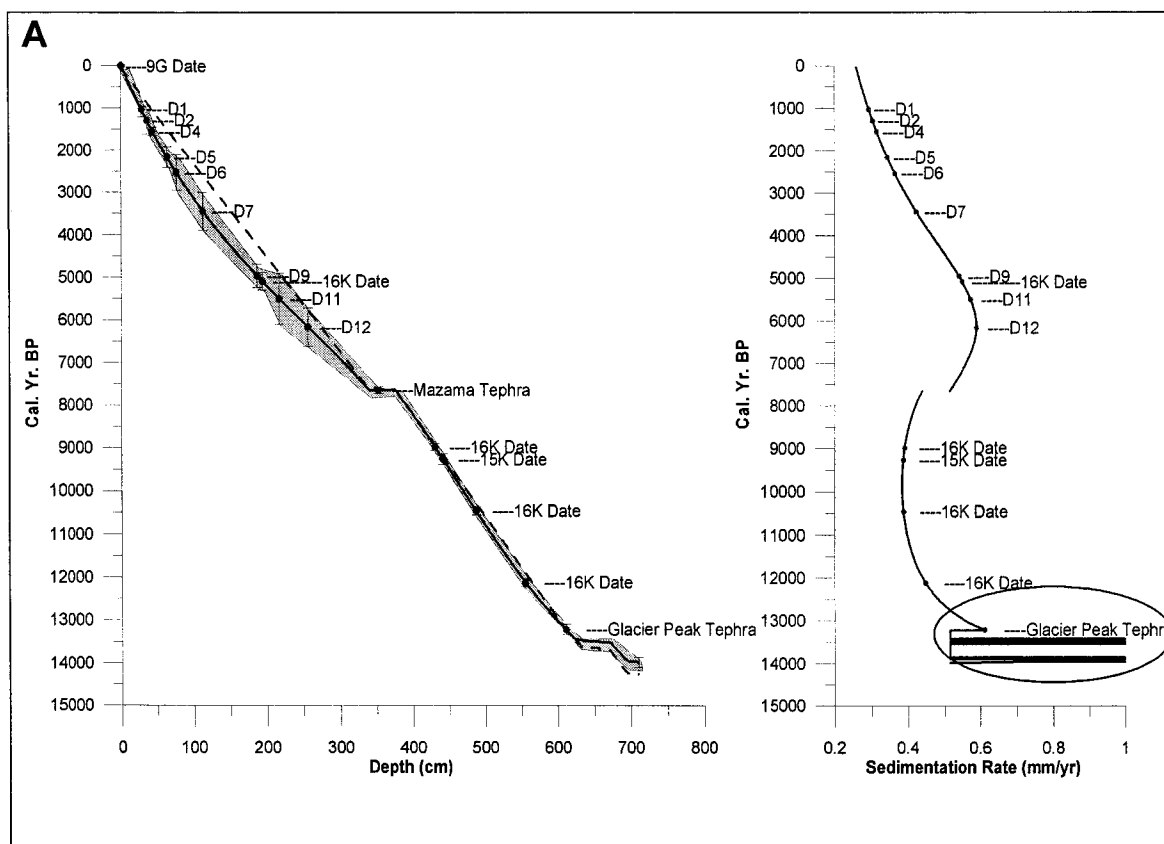
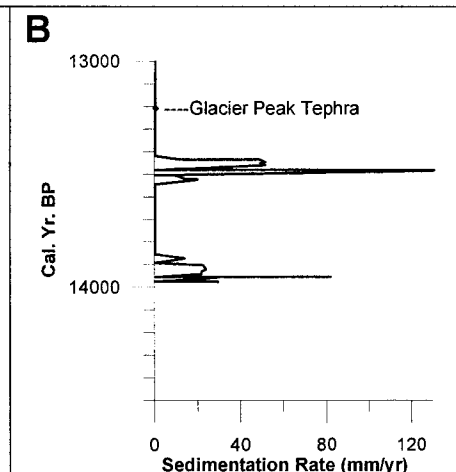
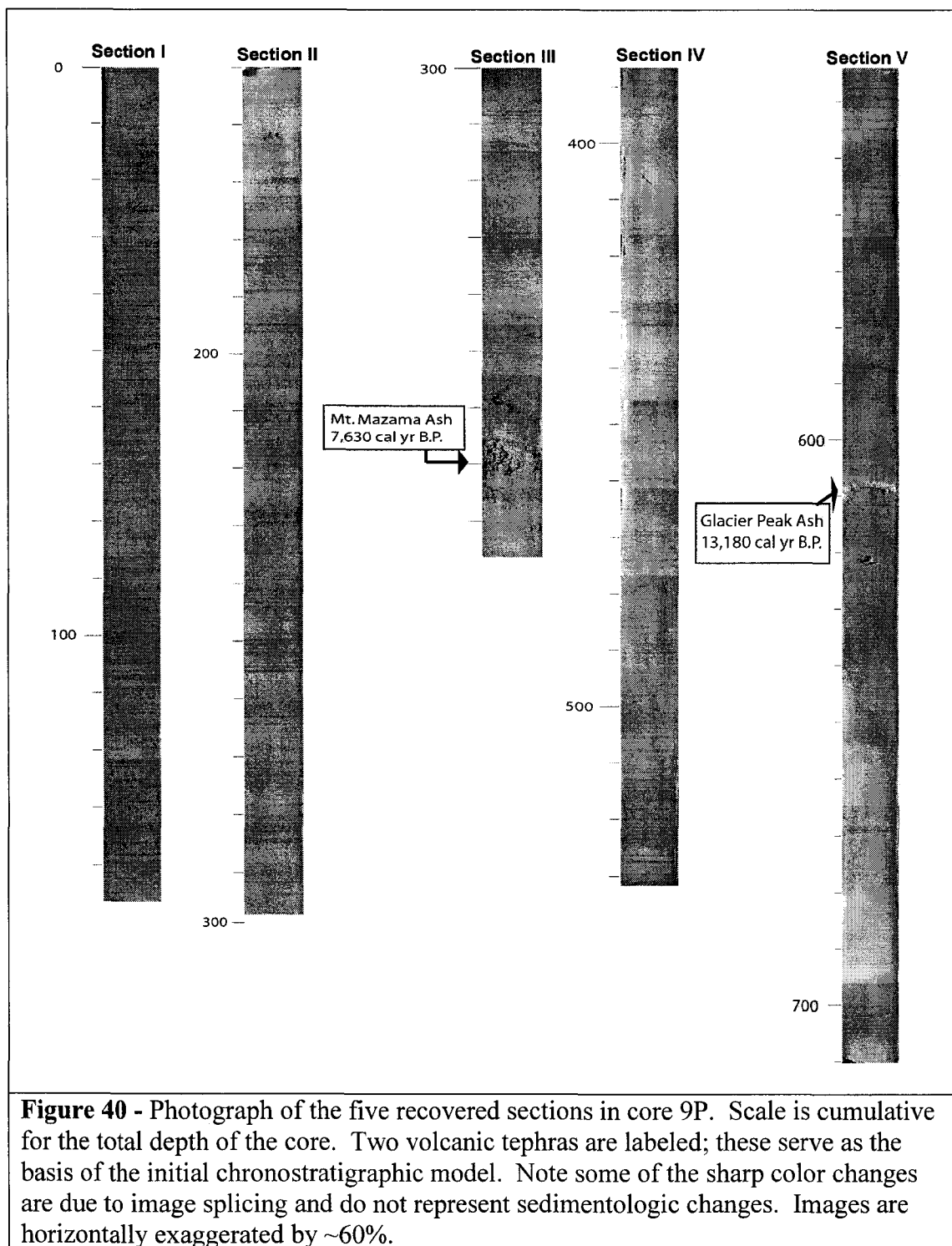
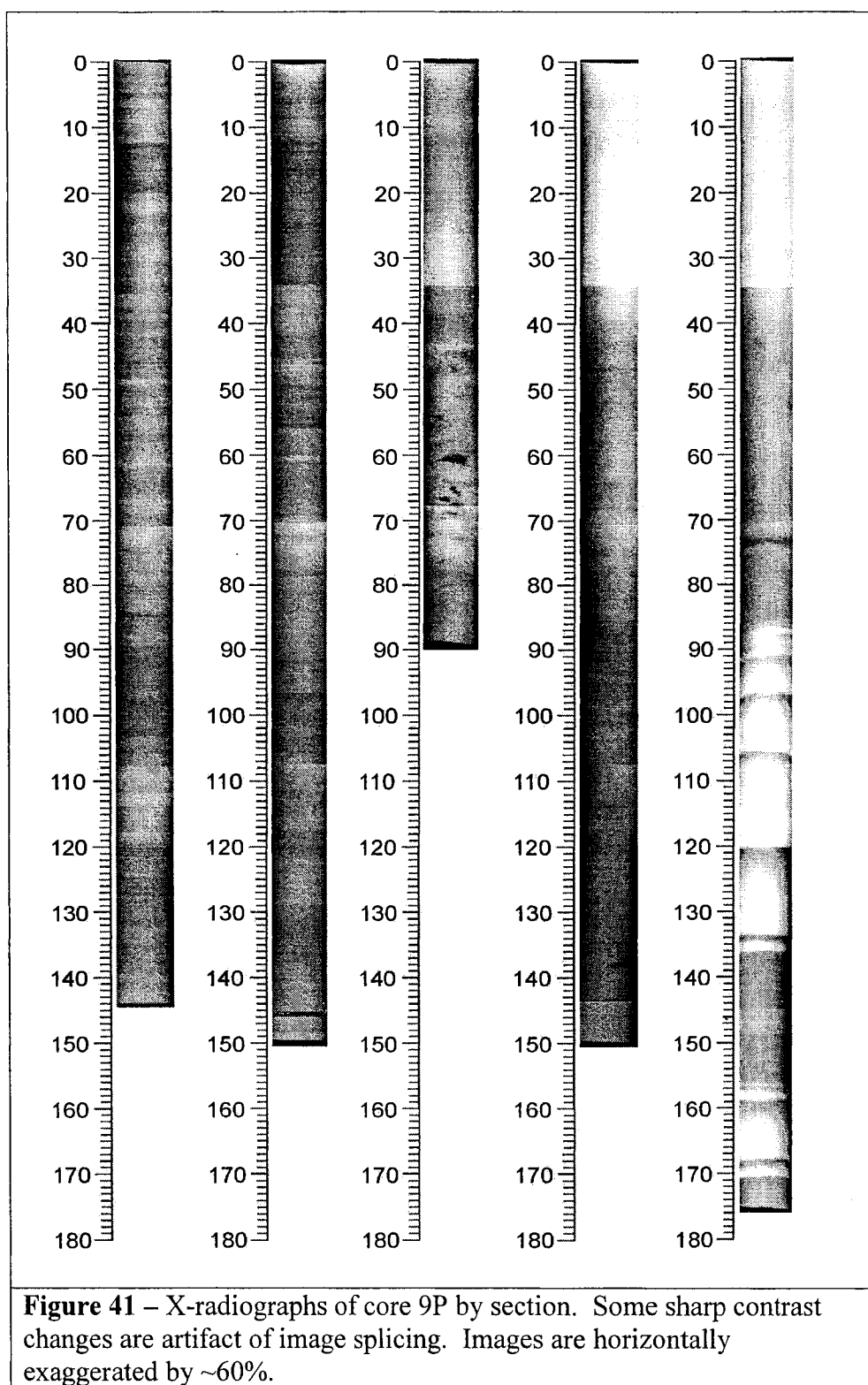


Figure 39 – (A) Final chronostratigraphic model for core FL-00-9P. Age model plotted for time vs. depth, points of correlation are shown with dots. Uncertainty shown with error bars for each tie point and with gray shadow range. Dashed line is initial age model. Graph on right shows sedimentation rate (unadjusted for water content) based on modeled timescale, bottom peaks truncated at right end of x-axis (oval). (B) Truncated portion of sedimentation rate graph (oval in A) showing sedimentation rate associated with upward-fining beds drafted to scale.



porosity, and sediment density. In June, 2003, U-channels were extracted from the archived core halves and sent to the paleomagnetism laboratory at the University of California-Davis for magnetostratigraphic measurements (Timmerman 2005; Sperazza et al. in prep.).





Core pucks from both the piston (working half) and gravity cores were subsampled for wet and dry analyses. Wet samples are needed for grain size, pollen, diatom, ostracode, and charcoal analyses. Approximately 2/3 of the each core puck has been either freeze-dried or dried in an oven at a temperature of 50 to 70 °C. Dry samples are utilized for geochemical analyses (TOC, TIC, elemental components), mineralogy, and discrete magnetic susceptibility measurements.

Sedimentary Facies:

A total of six sedimentologic facies are present in core 9P. Each was described based on initial core descriptions, photographs, and X-radiographs (Figs 40, 41 and Appendix C). Facies units are differentiated and described on the basis of changes in grain size, bed thickness, and abundance of black sulfide bands, presumed consist of mono-sulfide compounds (Fig. 42). The core 9P facies are described as follows:

Tep - The sedimentologic unit most visible in the core are two tephra located at 340-375 cm (Mt. Mazama, Crater Lake tephra) and at 610-611 cm (Glacier Peak, G tephra). The tephra are coarse grained (D_{50} 18 to 35 μm), and light in color ranging from 5/1 2.5Y to 6/1 2.5Y based on the Munsell Soil Color Chart. The upper tephra (Mazama) contains four beds of ash separated by deposits fine-grained sediment with faint laminae. These separate Mazama deposits likely represent tephra slumps due to rapid deposition on a subaqueous slope (Sperazza et al. in prep.).

Ufb - This sedimentologic unit consists of 13 coarse-grained (up to 18 μm), sharp-based upward fining deposits located between 622-671 cm and 691-710 cm. The individual upward-fining beds are cm- to dm-scale, with the thickest bed $\sim$ 14 cm. The beds are in two groupings separated by 20 cm of fine-grained hemipelagic mud (Hp4, see below). These upward-fining beds are believed to represent glacial flood deposits associated with Flathead Lobe retreat (Sperazza et al. 2002; Hofmann 2005).

Hp1 - Fine-grained (typically between 3.0 and 5.0 μm) hemipelagic lacustrine beds with evenly spaced surface laminae from mono-sulfides and evenly spaced mm-scale laminae in X-radiographs. Mono-sulfide beds rapidly oxidize and are lost visually within a few hours of splitting the core. In this facies, most surface laminations are composed of the black bands presumed to be mono-sulfides; mm-scale laminations are barely visible or not seen. However, sub-cm laminations are imaged in the X-radiographs, due to density differences in the sediment. The preservation of fine lamina in the X-radiographs demonstrates the lack of post-depositional sediment disturbances due to coring or bioturbation.

Hp2 - Very-fine grained (typically between 2.0 and 3.0 μm) hemipelagic lacustrine beds with surface mono-sulfides laminations evenly spaced $\sim$ 1 cm apart and evenly spaced $<$ 1 cm lamina in X-radiographs. Found in two intervals between 60 to 95 cm and 108 to 142 cm, with a color of 5/2 10YR.

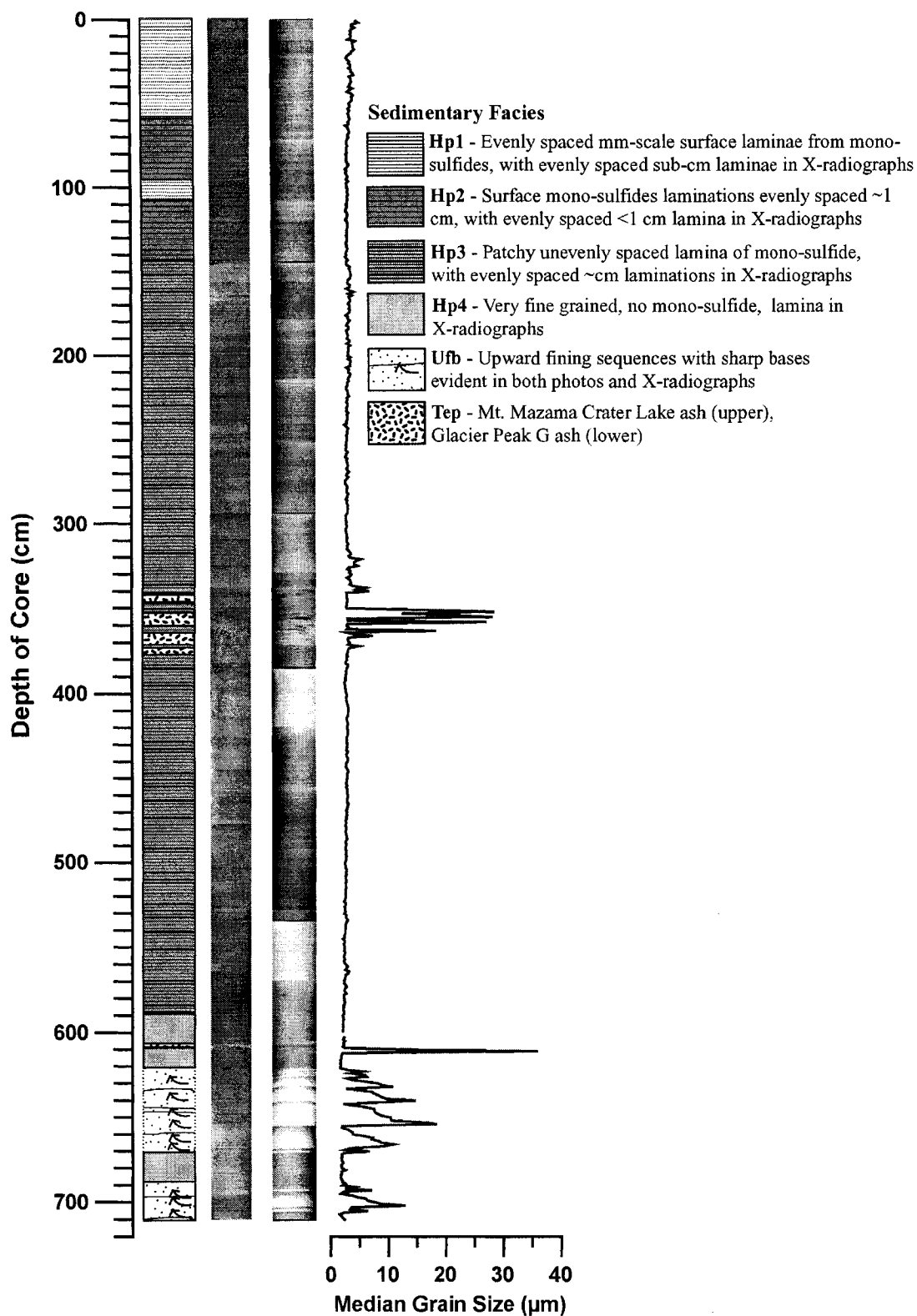


Figure 42 – Flathead Lake core 9P showing composite description of sedimentary units, Core photograph, X-radiograph, and grain size.

Hp3 - Very fine-grained (typically between 2.0 and 3.5 μm) hemipelagic lacustrine deposits with patchy unevenly spaced mono-sulfide lamina and evenly spaced ~cm laminations in X-radiographs. Massive hemipelagic deposits from 143 to 588 cm, interrupted only by the Mazama tephra, with color grading upward from 5/2 7.5YR to 5/2 10YR.

Hp4 – Very fine grained (typically < 2.1 μm) hemipelagic lacustrine deposits with no mono-sulfide beds, and only weakly expressed sub-cm lamina in X-radiographs. This facies is only found at the bottom of the core, between the upward-fining beds and only up to 588 cm. Beds have a color of 5/2 7.5YR.

Core description sheets for core 9P provide additional information about core color, bedding, and other initial observations. These sheets can be found in Appendix C, however the sheets for section I and II have been lost.

Proxies:

Since the direct measurement of past climate conditions is not possible, proxies are utilized to indirectly provide insight into climatic variability. Proxies however only measure the product of climate change, such as weathering rates, salinity, species adaptation, or compositional changes (Parrish 1998; Cronin 1999; Cohn 2003). The individual proxy may be recording changes within the lake (authigenic or endogenic) and/or changes derived from the watershed (allogenic or detrital) (Boyle 2001).

Additionally, most proxies can be influenced by more than one environmental change. For example, changes in types of tree pollen reaching a potential core site are affected by long-term variations in precipitation in addition to variations in temperature (Bartlein et al. 1998a). Thus, most paleoclimate studies utilize a multi-proxy approach to climatic reconstructions in order to discern simultaneous changes of multiple conditions (e.g., Mayle and Cwynar 1995; Andrews and Giraudeau 2003).

In paleoclimate research no specific set of proxies are universally used in all settings, although pollen and isotope-based studies are gaining popularity (e.g., Dansgaard 1987; Anderson 1993; Grootes 1993; Whitlock et al. 1993). The result is an assortment of proxy suites that make direct study-to-study comparisons difficult. One possible reason for the variations in the proxy suites utilized at different localities may be that not all proxies work in all settings, although this rarely if ever mentioned in the literature. The effectiveness of time-series proxy data from lakes can be influenced by topography and geology in the lake watershed, as well by the preservation of the proxy record within the lake sediments. Additionally, a researcher's expertise, available equipment, and funding constraints can influence proxy selection.

Proxy data archived in the sediments of small, closed lake systems frequently have been used to reconstruct regional and global climate histories through the establishment of multi-proxy time-series data sets (Mayle and Cwynar 1995; Campbell 1998; Yuretich et al. 1999; Andrews and Giraudeau 2003). In contrast, fewer such studies are conducted

on large, open lakes because of difficulties associated with understanding these more hydrologically and limnologically complex systems and the perception that sedimentary archives will be less sensitive to high frequency climate change because of the integration of the proxy record across the watershed.

In this study, I sought to examine the paleoclimatic potential of a set of proxies in a large lake setting situated in the northern US Rocky Mountains. The starting point of the research was an examination of the published literature to identify what proxies have been used and what potential climatic variations could be elucidated. The goal was to examine a broad spectrum of proxies, quantify their uncertainty, and assess their potential for climate reconstructions. These goals were necessarily constrained by available expertise, access to analytical equipment, and funding. Below I discuss the proxy groups investigated with respect to: 1) background review of past paleoclimate use in the literature, 2) data collection methodology employed in this study, 3) uncertainty calculations, and 4) potential as a climate indicator for Flathead Lake.

In this paleoclimate proxy analysis I collected data from 3 proxy categories: grain size, mineralogy, and carbon/nitrogen. From these analyses a total of 36 individual time-series proxies (26 measured directly and 10 calculated, typically as ratios of two proxies) were available for climatic consideration. All proxy data are presented as figures in Appendix D and in digital form in Appendix F.

Grain size

Background - The grain size of transported sediment is directly affected by the hydrologic competency of the transporting current. Transport of sediment has been correlated to flow velocity; a doubling of flow velocity can displace particles up to 64 times larger (Brooks et al. 1991). Therefore, variability of grain size recorded in lacustrine sediments may reflect changes in sediment load and thus river discharge, as shown in Pine Lake, Alberta, where coarse-grained sediment layers were correlated to periods of high streamflow (Campbell 1998). There, grain size increases were determined to be a function of the short residence time of the water within the lake and transport of the clay fraction out of the system during periods of increased through-flow (Campbell 1998; Campbell and Campbell 2002).

Alternatively, in the Mediterranean region of Europe Harrison and Digerfeldt (1993) found that increases in sediment grain size were caused by erosion of exposed lake sediments during lake-level lowstands. This climatic interpretation was strengthened by temporal correlation of lake level changes to other lake systems in the region (Harrison and Digerfeldt 1993), although lake level changes may also have physical, non-climatic origins, such as spill point down-cutting or basinal changes due to tectonic activity (Digerfeldt 1986). Grain size variability within a time-series data set may also be influenced by post-fire basinal erosion (MacDonald et al. 1991) or eolian dust transport (Porter 2000).

Methods - Traditional measurements of sediment grain size have employed settling tubes or pipette technology based on Stokes Law (Stokes 1880; Beuselinck et al. 1998; Baoping et al. 2002). However, these traditional methods have had a margin of error that can exceed 40 percent (Horiuchi et al. 2000; Wagner et al. 2000). The recent technology of laser diffraction has greatly reduced the error in grain size measurements, therefore expanding the value of grain size as a potentially sensitive paleoclimate proxy (Buurman et al. 1997; Beuselinck et al. 1998; Rawle 2000). In this study we analyzed grain size of core FL-00-9P on a Malvern Mastersizer 2000 with a Hydro 2000MU pump at 1 cm intervals for the entire 7.1 m core. All samples for core 9P were taken perpendicular to the depositional bedding using the direct-aliquot method, as described by Sperazza et al (2004), to assure a representative sample. Reported results are the average value of 3 measurements, totaling 36,000 pulses on the laser diffraction. In this study we report measures of grain size commonly used in climate studies. These include median grain size (D_{50}), and standard fine-grained sediment descriptors of percent clay, silt, and sand (Boggs 1995). More detailed discussions of laser diffraction for sediment grain size can be found in de Boer et al. (1987), Beuselinck et al. (1998), and Sperazza et al. (2004) and are not covered here.

Uncertainty - Methodological measurements of grain size by laser diffraction following those established by Sperazza et al. (2004) have an uncertainty of ~5% at 2 sigma. In this study we utilized two quality controls during the acquisition of grain size data. The first quality control protocol was that samples were measured three times, with each measurement collecting data from 12,000 laser pulses. The three measurements were

evaluated and accepted if the variation in median grain size was less than 5%. Samples not within this tolerance were rerun or, occasionally, reported as the average of only two measurements. Discrepancies in the three measurements were rare. Possible sources for inconsistency among the three individual measurements might include: 1) introduction of an air bubble during one or more of the measurements, 2) differential measurement of a few coarse grains, or 3) an unexplained machine spike.

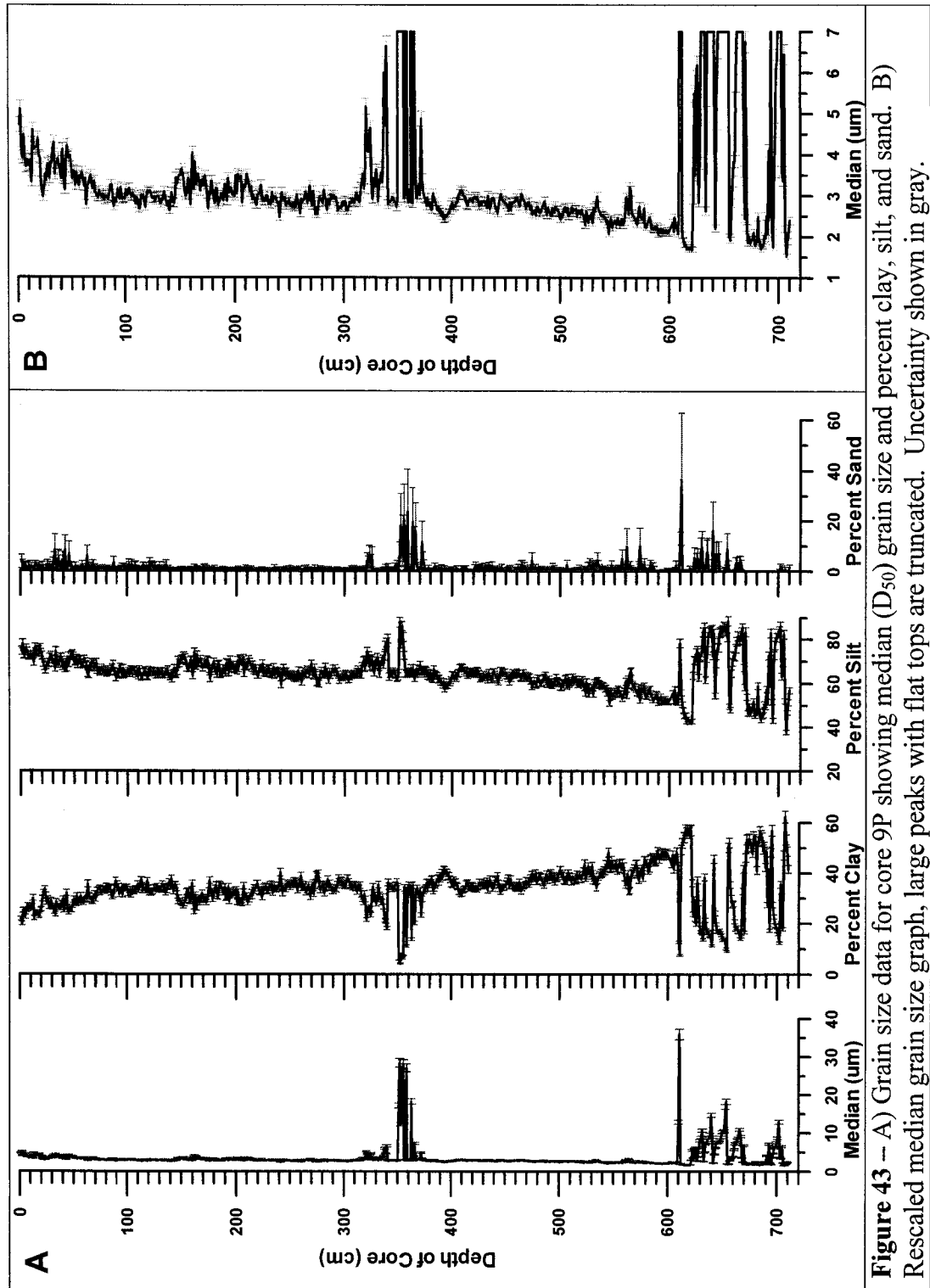
The second quality control protocol was that I assessed uncertainty by measuring replicate sediment samples every ~15 cm over the length of the core. A total of 38 replicate samples were analyzed to calculate grain size uncertainty for core 9P. Total uncertainty is composed of two components; percent difference of the replicate samples and two standard deviations (St. Dev.). Table 8 shows the components and total uncertainty for those measures report herein, along with some additional percentile measures. Uncertainty of median grain size (D_{50}) is comparable to reported methodological values by Sperazza et al. (2004).

Measure	D₁₀	D₂₀	D₅₀	D₈₀	D₉₀	%Clay	%Silt	%Sand
% Difference	2.01%	2.01%	2.26%	3.52%	6.09%	2.36%	1.27%	47.50%
2 St. Dev.	2.55%	2.72%	3.20%	5.69%	12.20%	3.28%	1.78%	94.77%
Total Uncertainty	4.56%	4.73%	5.46%	9.22%	18.29%	5.63%	3.05%	142.27%
Table 8 – Grain size uncertainty calculated for core FL-00-9P. Percentiles show as D_x measures, percent divisions based on Boggs (1995).								

Coarser size measures (D_{80} , D_{90} , and %Sand) have higher uncertainty values due to the very fine-grained nature of the core 9P lacustrine sediments and the presence of relatively few sand size grains.

Results and Discussion - Grain size data from core 9P reveal a series of cm- to dm-scale upward fining sequences from the bottom of the core (710 cm) to 622 cm, with the exception of the interval from 692 to 672 cm (Fig. 43). The thickness of the upward fining sequences ranges from 8 to 10 cm, some possibly being truncated by the overlying sequence. Above the uppermost upward fining sequence (621 cm) median grain size progressively increases from $< 2 \mu\text{m}$ to $\sim 4.5 \mu\text{m}$ just below the top of the core.

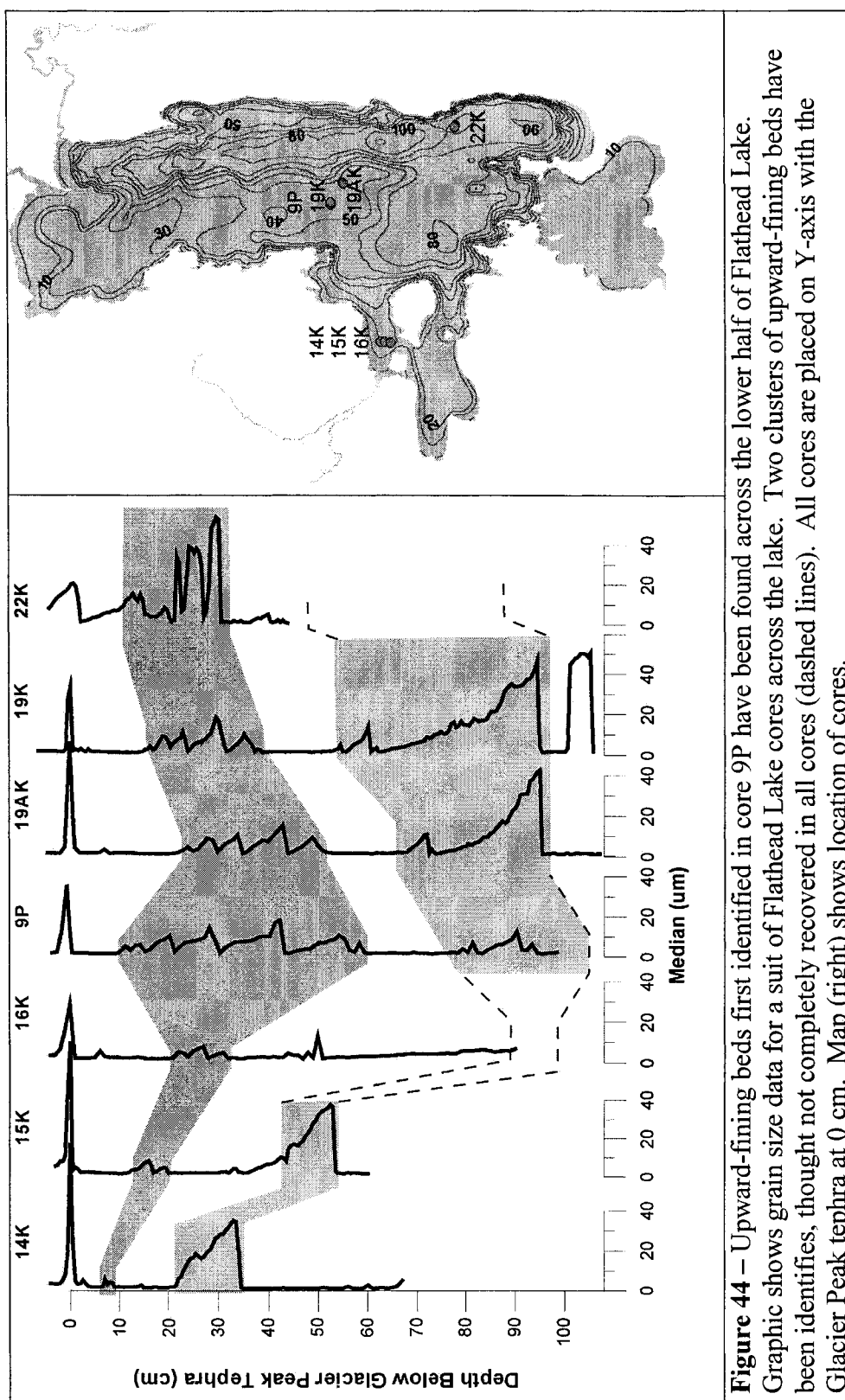
Sediments gradually coarsen upward over the length of the core above 621 cm, but are interrupted by the two relatively coarse volcanic ashes between 611-610 (Glacier Peak; $D_{50} \sim 35 \mu\text{m}$) and 375-341 cm (Mount Mazama; $D_{50} \sim 28 \mu\text{m}$). The upward coarsening nature of the core is within the measured uncertainty, however at a few intervals the change in grain size (coarsening or fining) is beyond calculated uncertainty and is therefore significant. Deviations from the general upward-coarsening (excluding the tephra) can be seen in median grain size, %Clay, and %Silt at 569-562, 539-531, 399-387, 325-320, 163-161, and 29-21 cm (Fig. 43). One of these deviations is particularly noteworthy; in the 399-387 cm interval %Clay smoothly increases to a peak of 41%, an increase of $\sim 35\%$ over the baseline above and below this interval. Also identified is a significant shift in the grain size records at a depth of 146 cm. The shift occurs 2 cm below a section break in the core (between sections I and II) and does not appear to be



related to the core section break. The magnitude of the shift is significant, increasing the average %Clay value by ~6 percentage points before continuing the upward coarsening trend. Grain size becomes more variable above 48 cm with apparent but statistically insignificant increases in the sand fraction (sand is within uncertainty).

Core 9P contains two distinct sedimentation styles (excluding the tephras); 1) coarse grained (up to 18 μm) upward fining centimeter scale beds and 2) very fine grained ($<4\mu\text{m}$) sub-cm scale laminae. The upward-fining beds are sharp-based and morphologically consistent with a waning flow, similar to sequences described and interpreted by Bouma (1962) to represent deposition by low density turbidity currents. Potential mechanisms for upward-fining beds at the bottom of core 9P include: low density turbidite sedimentation due to flood events (Miall 1990) or seismically-produced resedimentation events. However, seismic reflection data from Flathead Lake do not suggest that a seismic event occurred at this time (Hofmann et al. 2006; Hofmann et al. In Press). In addition, analyses of grain size from cores recovered in 2003 demonstrates that the upward-fining beds are a lake-wide feature, currently identified to occur across $\sim 180 \text{ km}^2$ of the lake floor. These observations suggest that the upward-fining beds are not local features (Fig. 44).

The beds are deposited in two clusters, separated by an interval of very-fine grain sediment. A reasonable explanation of the upward-fining beds is the release of ice or sediment dammed bodies of water. These swift releases of water would occur as dams of



ice or sediment failed or were downcut by streams that drained proglacial lakes that may have occupied tributary valleys upstream of Flathead Lake. The valleys east and northeast of Flathead Lake are numerous and likely contained proglacial lakes. In addition to existing glacially scoured lakes (such as Lake McDonald), evidence exists for pro-glacial lakes in the Nyack Valley (Nate Harrison 2004, per. comm.), Star Valley (Smith 2004), Swan Valley (Locke 1995), and in the Salish Mountains (Smith et al. 2000). Support for this interpretation also is found in the upper Flathead Valley itself, where the upper Flathead River has terraced and downcut glaciolacustrine sediments by more than 15 m. The anomalously fine-grained interval between the turbidites may represent ice lobe stagnation and lake level stability due to temporary climate amelioration. Alternatively, it may represent the establishment of a short-lived sediment trap behind one or more of the recessional moraines north of Flathead Lake (Smith 2004) and below the retreating ice lobe.

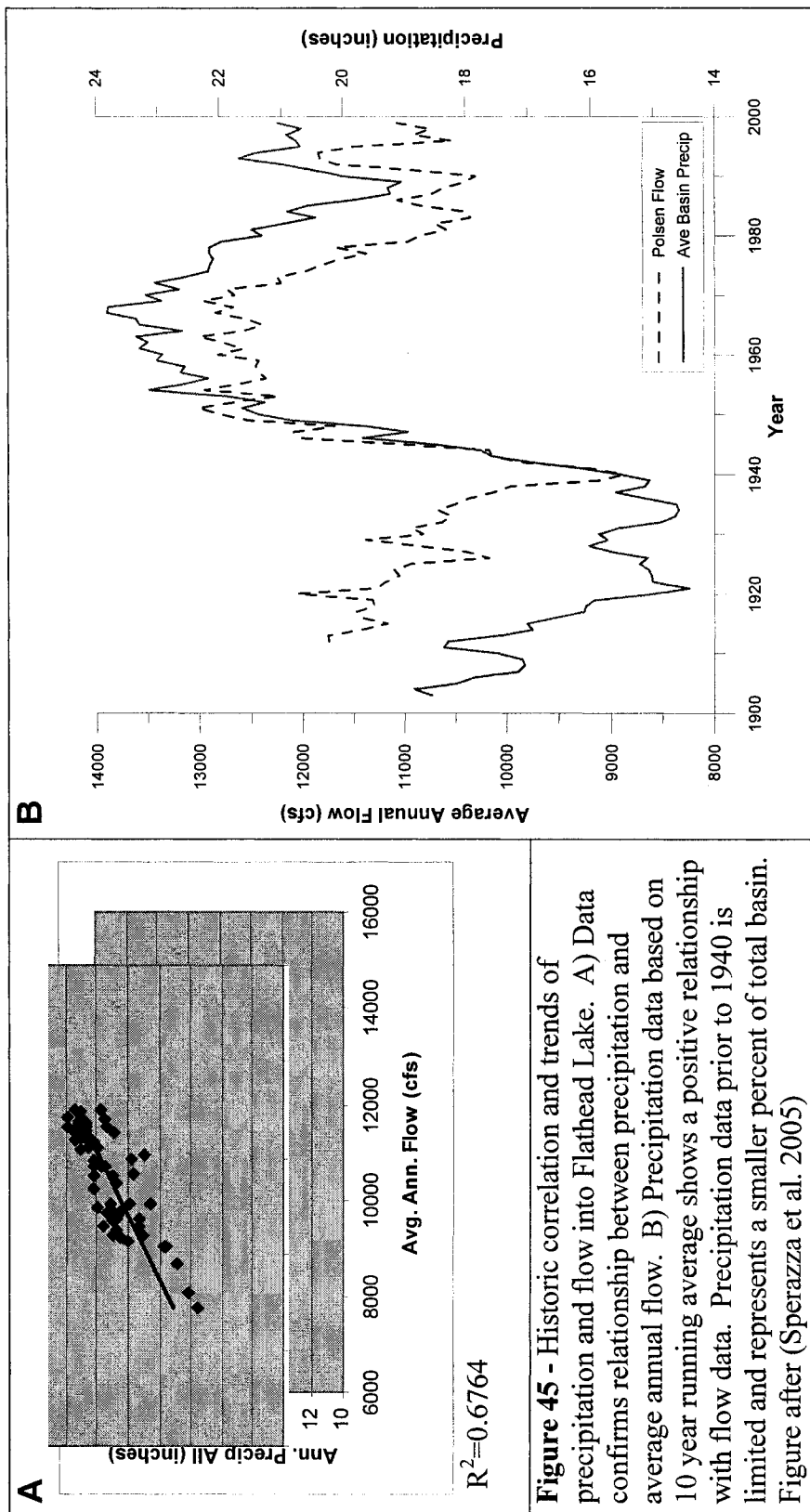
Upcore, the very-fine grained sediments contain no evidence of traction transport, such as ripples, and are likely deposited primarily by suspension settle-out. Two possible mechanisms with paleoclimatic implications that have been proposed for the interpretation of hemipelagic grain size variability include: 1) lake lowering and 2) adjustments of sediment delivered to the lake as a result of changes in flow (i.e., Digerfeldt 1986; Meyers et al. 1993; Campbell 1998)

The hydrology of Flathead Lake currently is dominated by alpine snowmelt. Moisture in the Flathead watershed is stored as snowpack and released in the spring as melt water.

Because ~ 90% of the sediment is delivered by the Flathead River, the spring melt water develops an annual sediment plume that can drift south over the length of the main lake body (~33 km). Based on traditional river transport dynamics, the resulting peak in annual discharge (Fig. 35) of the Flathead River should carry coarser sediments (Brooks et al. 1991; Boggs 1995) and may provide a potential proxy recorder of paleoprecipitation (Campbell 1998; Campbell et al. 1998). However, the duration and magnitude of melt water discharge into the lake also is influenced by the rate of temperature increase during the spring, cloud cover, and spring rainfall.

Modern observations support a correlation between high discharge events, maximum winter snowpack, and large sediment plumes transported southward through the lake system (Moore et al. 1982). Annual precipitation is strongly correlated ($R^2=0.6764$) with flow gauge data from the Flathead River (Fig. 45A) and tracks well with historical precipitation trends (Fig. 45B, Sperazza et al. 2005). Data shown in Figure 45 are based on 10 year running averages; precipitation data are from weather stations predominately located on valley floors with varying record durations. Prior to 1940 available station data are poorly maintained and represent a smaller fraction of the total precipitation in the drainage basin, but still track flow data (Sperazza et al. 2005).

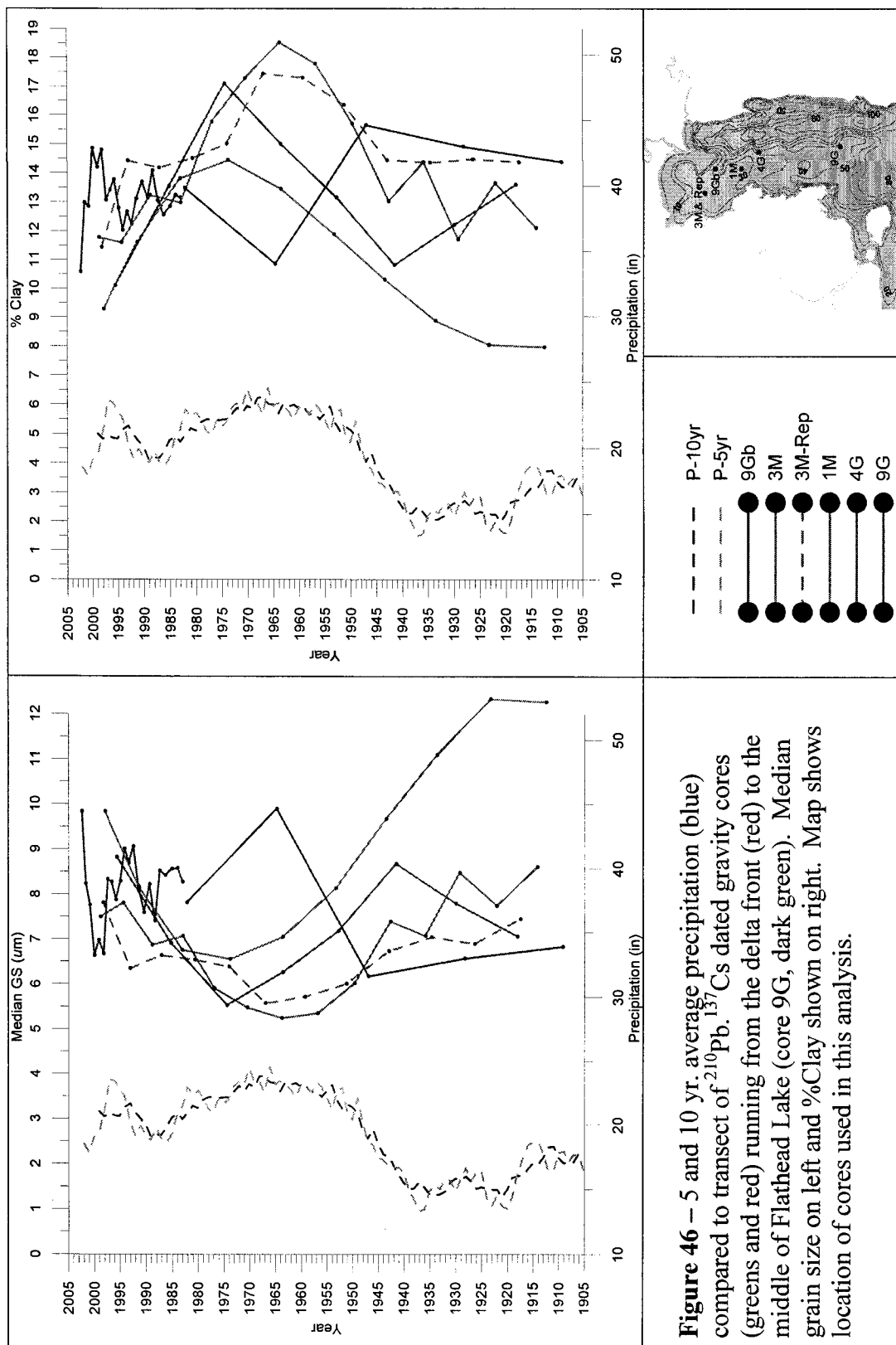
Flathead Lake has a short residence time (~2 years) (Hofmann 2005; Sperazza et al. 2005) and a dominant inflow source, similar to Pine Lake, Alberta, and could be used to test the grain size response to changes to precipitation, as proposed by Campbell (1998). Grain size data from six gravity cores with radiometric $^{210}\text{Pb}/^{137}\text{Cs}$ dating control were

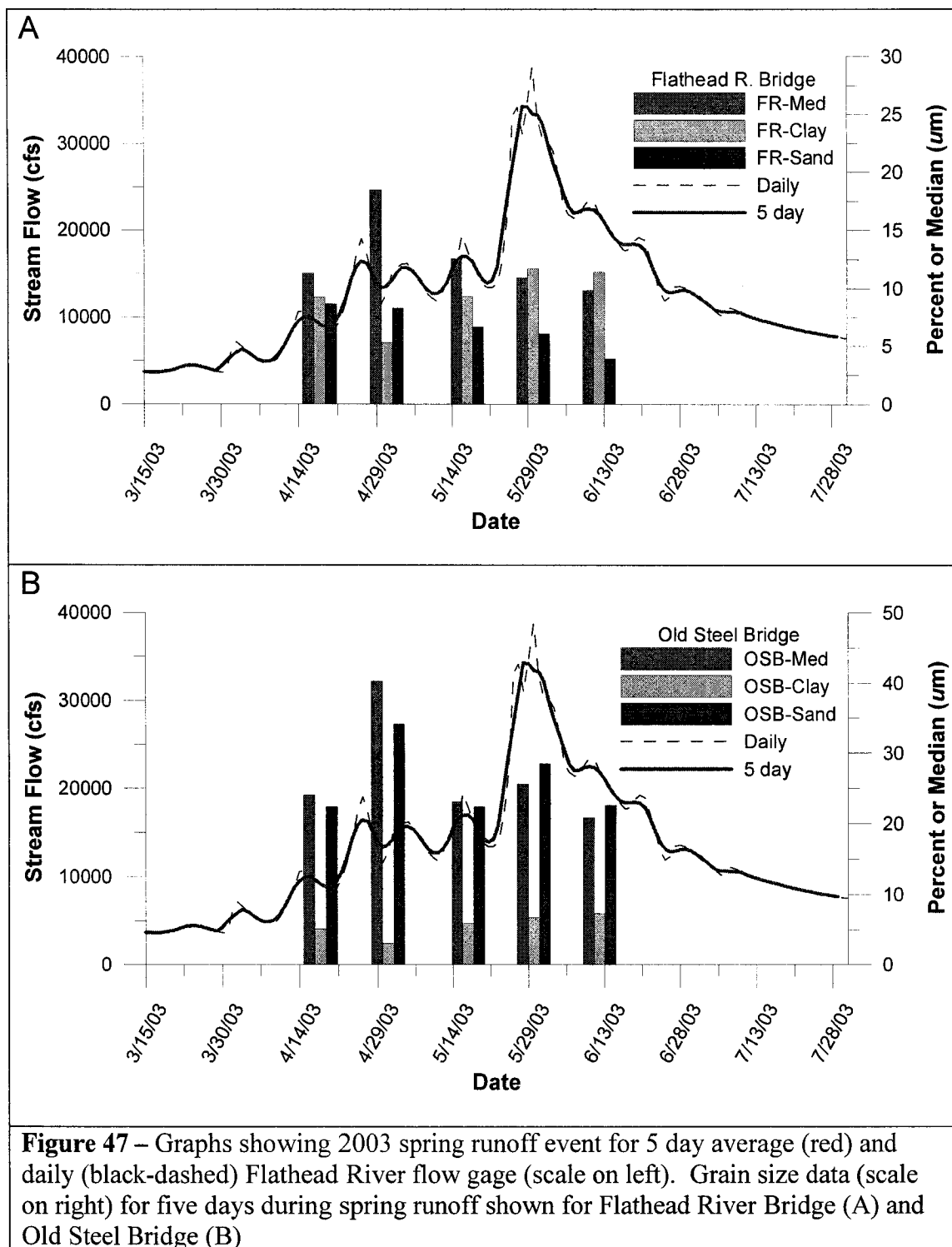


examined and compared to historical precipitation data. The cores were recovered between 3.8 and 18.8 km south of the Flathead River mouth. Because of the reduction in sedimentation rates away from the sediment source, the cores have a different number of grain size data points over the 100-year historical record. Campbell (1998) proposed a mechanism whereby hydrologic flow due to increased precipitation exported a larger fraction of the clay size grains out of the system, producing a period of partial sediment bypass and effectively increasing the overall grain size of the sample.

If the model Campbell (1998) proposed for Pine Lake also applied to the Flathead Lake system, median grain size should increase during periods of intensified precipitation. However, most Flathead gravity core data appear to show decreased median grain size and higher %Clay size grains during times of highest precipitation in the historical record, as defined by 5 and 10-year average precipitation data (Fig. 46). The possible exception is core 9G, 18.8 km from the Flathead River, which shows a general agreement with Campbell's (1998) hypothesis, although data points for core 9G are limited due to the generally low sedimentation rate > 18 km from the river mouth.

Further evidence against Campbell's (1998) hypothesis as an analog to Flathead Lake is grain size data collected from the Flathead River in the spring of 2003. These data were collected at two locations, one near the head of the delta (Flathead River Bridge) and the other ~39 km upstream (Old Steel Bridge). Grain size of sediments suspended in the river water were sampled and measured five times over the course of the spring runoff during 2003 (Fig. 47). Approximately 5 gallons of river water were hand-sampled and





transported to UM. Samples were allowed to settle one week and excess water was siphoned off to a volume of < 450 ml. Samples were completely introduced and measured on the Malvern Mastersizer using procedures established by Sperazza et al. (2004). Correlations of grain size changes with river flow data are very poor, the best comparison being flow to %Clay with a $R^2 = 0.34$. These data, while limited, suggest that precipitation has not been the dominant control on sediment grain size in Flathead Lake during the previous century.

Broadly, variations in grain size have been used to interpret fluctuations in lake level. Some investigators have used grain size as an indicator of proximity to sediment source during lake level fluctuations (Heimann and Braun 2000; Andrews and Giraudeau 2003; Curtin et al. 2003; Zybala et al. 2003), whereas others have suggested that grain size varies as a function of erosion and remobilization of exposed shoreline sediments during transgressive and regressive phases (Harrison and Digerfeldt 1993; Venczel 2005). The use of grain size as a proxy for lake level fluctuation has been strengthened through correlation with other proxy data. For example, lower lake levels were interpreted from Summer Lake, Oregon based on changes in the ratio of %silt and %clay and the correlation of these data with TOC (Negrini et al. 2000; Zic et al. 2002).

Lake level fluctuations of large lakes mostly have been attributed to structural changes of the basin or down cutting of the spillway. However, in a hydrologic model of lake level changes in Lake Malawi, Owen et al. (1990) demonstrated that small changes in the

precipitation / evaporation ratio could appreciably lower lake levels. For example, they found a 10% change in lake volume and 150 m drop in level was produced by only a 30% decrease in precipitation. The decrease in lake level elevation, which was estimated through modeling to have occurred over 200 years, did not coincided with an appreciable change in the percentage of carbonate sedimentation in the lake. These observations suggest that the concentration of carbonate ionic species had not reached a supersaturated level (Finney and Johnson 1991).

In the Flathead Lake basin, shoreline terraces from higher lake stands are evident on the Polson and Big Arm moraines where they represent temporary highstands by a proglacial lake that occupied the valley during deglaciation (ancestral Flathead Lake, Smith 2004). Although the ages of these terraces are unknown, they can be explained by physical down cutting of the spillway through the poorly consolidated moraine sediments.

In Big Arm Bay, an embayment with less bathymetric relief than the rest of the lake, seismic reflection lines exhibit a series of onlap and toplap stratigraphic geometries (Kogan 1980; Hofmann et al. 2003; Hofmann et al. 2006) that reflect lake level fluctuations. Seismic onlap geometries such as these suggest erosion due to lake level lowering and commensurate lowering of wave-base elevations, followed by redeposition during transgression of the lake level (Mitchum et al. 1977). A series of cores (FL-03-14K, FL-03-15K, and FL-03-16K) recovered along one of the seismic lines in 2003 confirms the presence of the most pronounced truncation surface in the seismic data

(Hofmann et al. 2003). Correlation of the seismic and core data in Big Arm Bay suggest that erosion associated with this surface occurred immediately after deposition of the Mount Mazama tephra (7,630 cal yr BP, Zdanowicz et al. 1999).

The surface elevation of Flathead Lake during this Early Holocene lowstand is estimated to be ~15 m below the current the bedrock-controlled spillway elevation, which sits on a ridge of Proterozoic metasedimentary Belt Supergroup rock approximately 6 km upstream of Kerr Dam (Levish 1997; Hofmann 2005; Sperazza et al. 2005; Hofmann et al. Submitted). No lower elevation spillway or alternative path for the inflowing Flathead River has been identified, and the seismic reflectors within the lake lack any offset at this time that might indicate a tectonic cause for the observed stratal geometries.

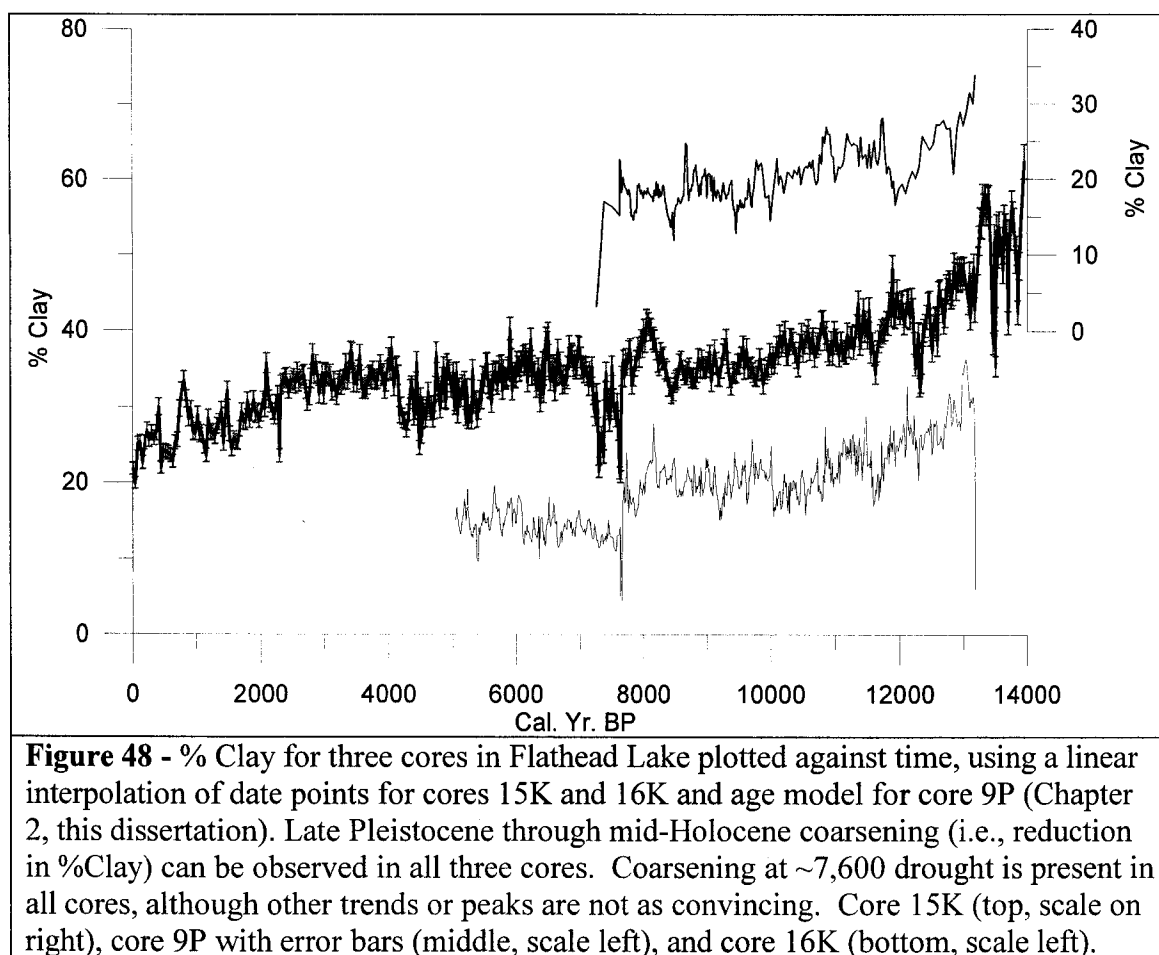
The most plausible explanation for the lake level lowering associated with the observed erosional surface at the top of seismic unit D (Fig. 8, p. 124, Hofmann et al. 2006) is climate change and a severe drought that reduced lake volume by an ~ 25%. Given these interpretations, it is necessary to explain the lack of carbonate deposition in the sedimentary record for this time period, because increased evaporative concentration of lake water would push the chemistry of the system in the direction of carbonate precipitation.

Significant lake level fluctuations have been reported elsewhere without carbonate precipitation (Johnson et al. 1996; Johnson et al. 1998). Geochemical modeling of Flathead Lake water chemistry in relation to ground and surface waters have

demonstrated that a rapid decrease in lake level would not result in precipitation of carbonate minerals (Hofmann 2005; Sperazza et al. 2005; Hofmann et al. Submitted). An independent confirmation of Early Holocene lake level lowering comes from Foy Lake, a small closed lake system in the watershed north of Flathead Lake. The Foy Lake seismic data contains onlap and toplap structures similar to, and temporally synchronous, with the Flathead Lake record (Colman 2003).

Rapid and severe climate change associated with the post-7.6 ka erosion surface is suggested by multiple grain size records from Flathead Lake cores (Fig. 43 and 48), although the presently available data does not require the interpretation that all grain size fluctuations observed in core records from Flathead Lake are controlled by lake level. In the early Holocene record from core FL-00-9P, some peaks or trends identified in core 9P are not present in the other core records (Fig. 48), suggesting that the entire system is not behaving in phase with respect to temporal fluctuations in grain size. I propose that the general upward-coarsening Holocene trend in median grain size from core 9P (Fig. 43) and corresponding overall decreasing trends in %clay from cores 15K and 16K (Fig. 48) are a function of reduced distance to the sediment source (river mouth) resulting from delta progradation, rather than a direct result of climate related lake-level fluctuation.

To explore this hypothesis, I analyzed sediment grab samples collected from a grid across Flathead Lake (Fig. 49C) and measured for grain size using settling tube methods (Moore et al. 1982). The results strongly suggest a relationship between grain size and distance



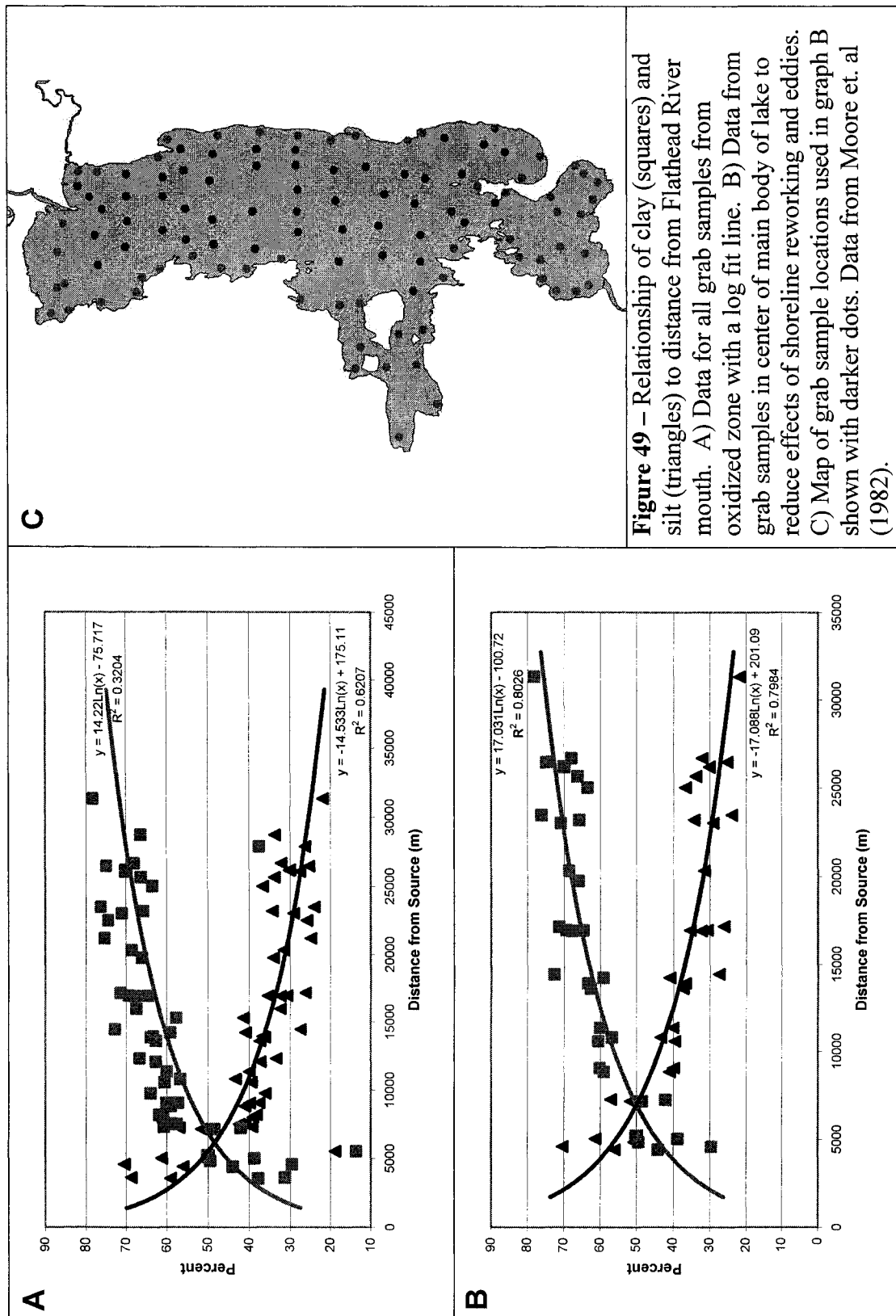
from sediment source (Fig. 49A). The correlation is strengthened by excluding samples located well away from areas affected by the annual spring sediment plume as well as samples collected along the shore which are prone to shoreline reworking and eddy effects (Fig. 49B).

In order to compare settling tube derived grain size results reported in Moore et al (1982) with grain size results collected through laser diffraction analysis of presently available cores from Flathead Lake, I attempted a replication study. Laser diffraction grain size data were measured from the core sediment layers that correlated to 1980 (the year of the

Moore et al. samples), based on available $^{137}\text{Cs}/^{210}\text{Pb}$ dating. Grain size distributions as determined from laser diffraction were recalculated to match the 4 μm clay/silt division of the original study (Moore et al. 1982). Whereas the laser diffraction results are limited, they do not show the same correlation between grain size and distance to the main sediment source (the Flathead River mouth) (Fig. 50). A possible explanation for observed differences between laser diffraction and settling tube data include the amalgamation of larger sediment samples (grab sample) during recovery and/or the higher uncertainty of the settling tube methodology (Moore et al. 1982; Sperazza et al. 2004). Other efforts to find a relationship between distance and grain size based on the cores recovered between 2000 and 2002 have yielded the same weak correlation shown in Figure 50. While grain size data from additional cores may clarify the influence of lake level on grain size distributions across the lake basin, I posit that the grain size record is buffered by overlapping controls that include lake level, sediment throughflow, and sediment distribution across the lake bottom. Therefore, I conclude that grain size data is only sensitive enough to reflect large hydrologic changes consistently across the lake.

Mineralogy

Background – Variations in mineral concentrations, ratios, and indexes have been shown to serve as proxies for variation in precipitation, weathering and lake productivity (Hay et al. 1991; Li et al. 1997; Ergin et al. 1999). However, paleoclimate interpretations rely in part on an understanding of the provenance of minerals that comprise lake sediments.



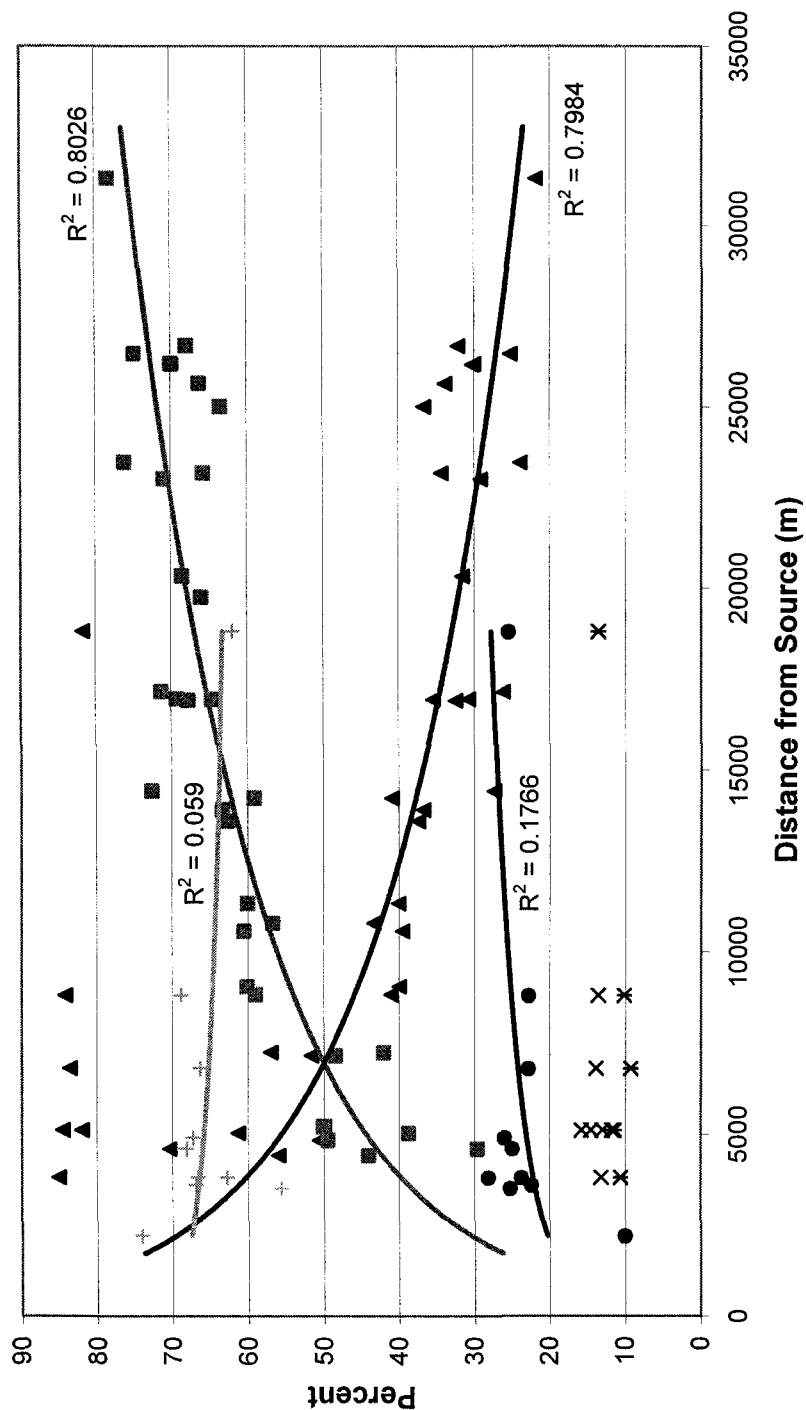


Figure 50 – Correlation of %Clay and %Silt with distance to source. Laser Diffraction grain size data is plotted with settling tube data. %Clay (diamonds) and %Silt (plus) laser diffraction data show little correlation with settling tube data (%Clay, squares and %Silt, triangles). Compared data is all for top oxidized layer and using clay fraction as <4 μm (Wentworth 1922). Also shown in black are laser diffraction data based on clay fraction <2 μm (Boggs 1995). Laser diffraction data is from $^{137}\text{Sc}/^{210}\text{Pb}$ dated cores, comparing the same 1980 interval used in the settling tube data, %Clay (X), %Silt (triangles on top), and top oxidized layer ~2001 (star).

Some minerals are truly detrital, whereas others are authigenic (Last 2001).

Understanding of the genesis of sediment contributions is particularly important when carbonate and clay minerals are used to infer paleoclimate. Although diagenesis of clay minerals can be substantial in older lake sediments, it has been reported as insignificant for paleoclimate interpretations in Pleistocene/Holocene sediments (Chamley 1989).

In this study, a total of 21 mineralogic proxies were developed either as percent concentration of individual minerals or mineral groups or as calculated indices from the Flathead Lake QXRD data. Of these 21 proxies, eight have been presented as indicators of paleoclimatic by other researchers and are reviewed herein.

Illite Crystallinity:

The crystal structure of the clay mineral illite is susceptible to weathering from increased moisture. During the weathering process, cations are removed from the illite structure and other clay minerals are produced as a secondary product (Barshad 1966). The illite crystallinity index has been utilized in lacustrine (Horiuchi et al. 2000; Fagel et al. 2003) and marine (Ehrmann 1998; Thamban et al. 2002) studies as a proxy for weathering intensity and an indicator of warmer, wetter climate conditions. A low illite crystallinity index indicates low or slow rates of weathering, suggestive of cooler drier climates (Horiuchi et al. 2000; Fagel et al. 2003) and more dominate physical weathering conditions (Chamley 1989; Ehrmann 1998). Conversely, a higher index indicated by a wider 10 Å peak on X-ray diffraction data, indicates reduced crystallinity and increased weathering (Chamley 1989).

Clay Minerals:

The clay mineral kaolinite and the ratio of kaolinite/illite clays also have been used as weathering proxies for changes in precipitation and humidity (Lauer-Leredde et al. 1998; Clayton et al. 1999; Foucault and Melieres 2000; Ruffell and Worden 2000; Gingele et al. 2001). Kaolinite develops in sediments that have been exposed to heavy chemical weathering in which cations are leached out, commonly in warm moist climates (Keller 1970; Yuretich et al. 1999; Fagel et al. 2003). Conversely, illite formation is prevalent under arid/cool conditions (Ergin et al. 1999). Given these general observations, researchers have suggested that the kaolinite/illite ratio can serve as a humidity indicator (Chamley 1989; Thamban et al. 2002). Chlorite minerals also are susceptible to chemical weathering and degradation from transportation (Biscaye 1965), and have been used to infer periods of physical weathering or glacial processes (Ehrmann 1998).

Biogenic Silica Concentrations:

Increases in diatom abundance and speciation (i.e. primary lake productivity) have been utilized by researchers to suggest periods of warmer temperatures in both marine and lacustrine environments (Wagner et al. 2000; Chebykin et al. 2002). In the Indian Ocean, biogenic silica concentrations in bottom sediment (a measure of diatom productivity) increased northward from the equatorial region after the last glacial maximum due to increasing temperatures and oxygenation of the waters (Bareille et al. 1998). Moreover, strong agreement between %opal (biogenic Si) and the GRIP2 $\delta^{18}\text{O}$ temperature proxy has been shown at Lake BasaltsØ, East Greenland (Wagner et al. 2000). Although

diatom productivity has been shown to correlate with temperature variations, it is also sensitive to other climatic conditions, such as duration and extent of ice and snow cover and nutrient loading (Colman et al. 2005; Mackay et al. 2005). Biogenic silica (also called biogenic opal) has served as a proxy for diatom abundance in many paleoclimatic studies (Bareille et al. 1990; Qiu et al. 1993; Bareille et al. 1998; Brauer et al. 1999; Talbot and Lærdal 2000; Fedotov et al. 2004). However, a recent study in Lake Baikal cautions against using only biogenic silica to infer lake productivity, because differential diatom preservation in the sedimentary record may not be climatically controlled (Battarbee et al. 2005).

Siderite Concentrations:

The mineral siderite has been suggested to indicate increased precipitation in forested basins with developed soils, due to mobilization of iron into the lake (Sifeddine et al. 2003). Siderite formation is favored in environments that are suboxic, non-sulfur bearing, and with available iron and low organic carbon concentrations (Berner 1981). However, siderite formation in the water column has also been suggested during warm and humid periods with abundant inflow of Fe and Si, which would then be preserved in anoxic bottom waters due to restricted turnover (Hsu and Kelts 1978), reduced sediments from organic decay (Wagner et al. 2000), or rapid burial (Williamson et al. 1998). The periods of siderite precipitation in the Black Sea that are inferred to represent heavy chemical weathering of a warm humid environment also are supported by palynological studies from the basin (Rajan et al. 1996).

Carbonate Mineral Species:

Carbonate minerals are heavily used as important paleoclimate proxies in saline and hyper-saline lacustrine settings (e.g., Valero-Garcés and Kelts 1995; Anderson 2001). The assemblage of precipitated carbonate minerals is sensitive to changes in temperature and water chemistry (Cohn 2003) and productivity of calcareous organisms (Dean and Megard 1993; Dean 1999). However, microbial activity also has been shown to contribute to carbonate precipitation in oligotrophic lakes, thereby complicating the paleoclimatic interpretation (Moore 1983). Additionally, authigenic carbonate can develop from ionic concentration and salinity saturation of the water column due to climate-driven lake lowering.

Understanding lake water balance controls is complicated and subject to many variables (Hofmann 2005). For example, in a hydrologic model involving lake level changes in Lake Malawi, Owen et al. (1990) demonstrated that small changes in the precipitation / evaporation ratio could appreciably lower lake surface elevations. They reported a 10% reduction in lake volume and 150 m drop in lake level with only a 30% decrease in precipitation. Nevertheless, according to (Finney and Johnson 1991), decreases in lake level, which was modeled to occur over 200 years, were not necessarily recorded in the sediments as calcite deposition if carbonate concentrations had not reached a supersaturated level.

Methods - Mineralogical data was collected at 10 cm intervals for the entire length of core 9P and at 5 cm intervals for that section of the core that brackets the Pleistocene/Holocene boundary (684-584 cm). In this study quantitative mineral determinations were calculated utilizing randomly-oriented sediment samples with an internal standard (Chung 1974) to measure bulk mineral phases and major clay mineral families (Środoń et al. 2001). Core samples with the internal standard (10%wt. zinc oxide) were precision wet ground, to achieve a narrow grain size distribution, in a McCrone Micronizing Mill. Mineralogic data were acquired by X-ray diffraction analysis on either a Bruker-AXS D5000 Diffractometer or a Bruker-AXS D8 Diffractometer. The diffraction scans used Cu K α radiation recording data over a range from 5 to 65 °2 θ with incremental steps of 0.02 °2 θ . Counting rate at each step was 4 s. Calculations of quantitative X-ray diffraction (QXRD) analysis followed the sample preparation methods and analytical techniques detailed by Środoń et al. (2001), and are not reviewed here. All QXRD analyses were performed at the Houston Research Center of Chevron Corporation.

Mineralogic indices are calculated values based on the graphical measurement of an X-ray diffraction peak width and/or height. In this study the illite crystallinity index was calculated by measuring the width of illite (001) peak, which is found at a d-spacing of 10 Å. The peak width is measured at half the distance between the peak maximum intensity and the baseline on the XRD pattern (Horiuchi et al. 2000). The other calculated mineral index is biogenic opal, which is measured at the cristobalite (101) peak or the maximum opal bulge height. Three different methods to calculate biogenic

opal have been described by Goldberg (1958), Eisma and Van der Gaast (1971), and Fagel et al. (2003). All three techniques reflect relative changes as compared to other values prepared by the same procedures. The simplest method is to record and compare the total counts at the maximum of the 4.04 Å (21.98 °2θ) peak (Goldberg 1958). Biogenic opal at the 4.04 Å peak base is measured over the baseline and multiplied by 20, as described by Fagel et al.(2003). However, for graphical presentation in this study, these data are shown without the multiplication factor. Eisma and Van der Gaast (1971) suggested measuring the maximum opal bulge at 26 °2θ above a baseline drawn straight between the base points at 17 °2θ and 46 °2θ. All three methods yield similar results (Appendix D), but with varying intensities. In this study the method described by Goldberg (1958) is used to assess biogenic Si as a paleoclimate proxy. All relevant calculations were performed using the digital XRD data and an Excel spreadsheet.

An important consideration when using mineralogy for paleoclimatic research is the determination of the mineral origin. Are the grains allogenic (detrital) or authigenic (Last 2001)? This determination was made by visually examining the mineral grains on a Hitachi S-4700 Scanning Electron Microscope (SEM) at The University of Montana, Department of Biological Sciences. The theory and operational details of the SEM can be found in Goldstein et al. (1981) and Dykstra (1992) and are not reviewed here. Mineralogic determinations also made use of elemental data collected on a Gresham Scientific Instruments, Energy Dispersive Spectrometry (EDS) Sirius Model 30, with Quartz X One (v.5) software. Dried sediment samples were gently split with a razor and then mounted on the stage with either silver glue or on carbon tape. Samples received

two coats of conductive metal (Au:Pd) for 30 s at 18 mA with a Pelco Model 3 Sputter Coater 91000. Additional detail of sample preparation for geologic samples can be found in Welton (1984).

In these SEM studies, I sought to characterize carbonate and clay mineral origins, because both mineral classes can be detrital or authigenic in this natural setting. I also wanted to find supporting data for the biogenic opal results calculated from the X-ray diffraction data (Goldberg 1958; Eisma and Van der Gaast 1971; Fagel et al. 2003). SEM samples were selected from core 9P based on the minerals of interest, so that high and low QXRD values could be examined (Fig. 51). One sample from core 9P (FL-00-9P-I-2) did not have direct mineralogic data and was examined to characterize modern sediment textures and compositions, to the extent possible. A few samples were examined from core 16K to characterize the Mt. Mazama tephra (FL-03-16K-II-145) and the deposits just above the tephra (FL-03-16K-II-130 and FL-03-16K-II-129).

Uncertainty –Środoń et al. (2001) reportedly achieved error margins of less than 2%wt for typical mudstone composition. This method compares favorably to other semi-quantitative methods that have estimated uncertainty of $\pm 5 - 10\%$ (Biscaye 1965). However, the accuracy for the QXRD methodology is based on artificial mineral mixtures with a limited number (2 or 3) of constituents (Eberl 2003).

In this study, I conducted an error analysis of the quantitative X-ray diffraction mineralogic assessment technique described above. Specifically, I reanalyzed 20 samples

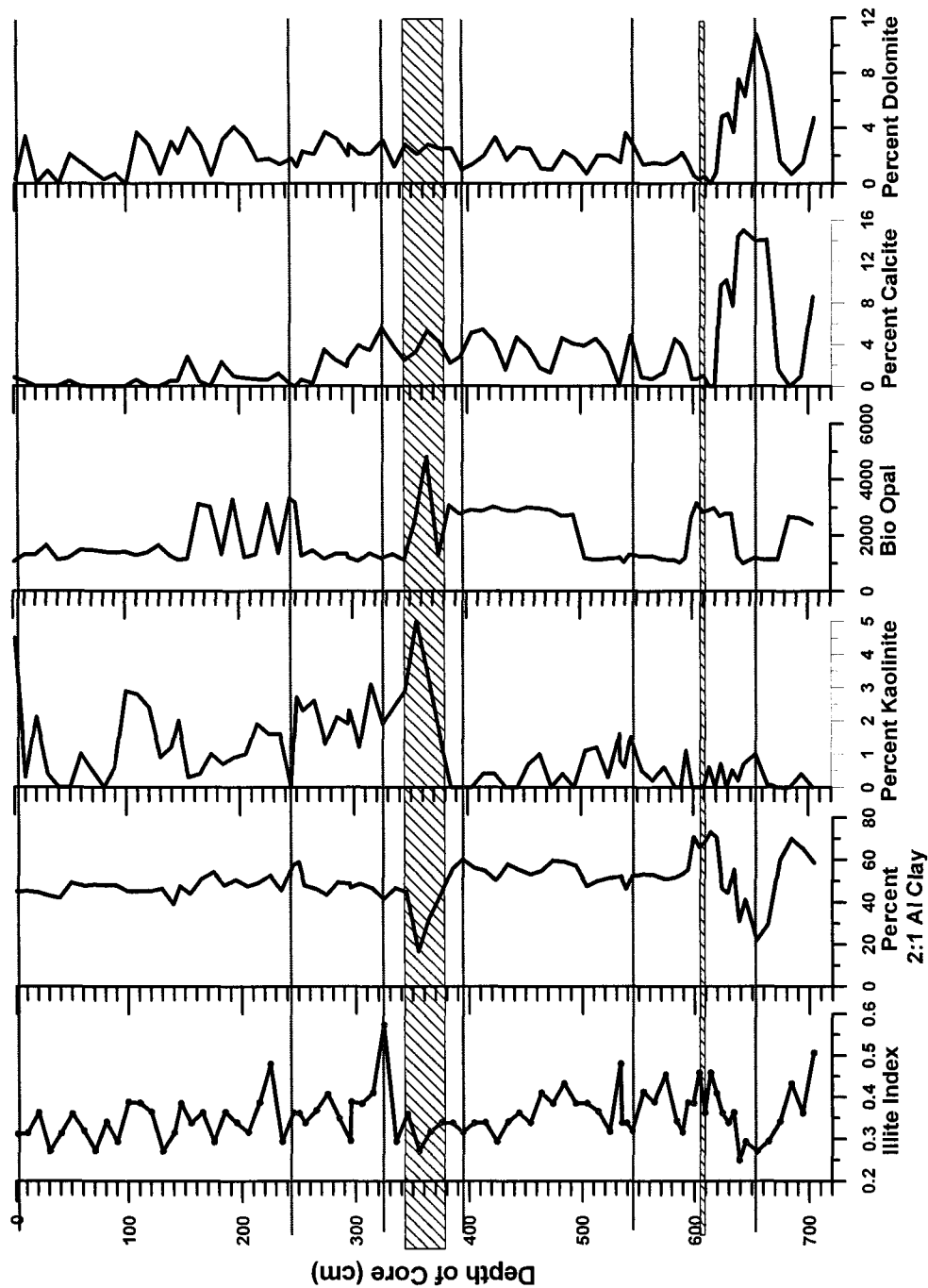


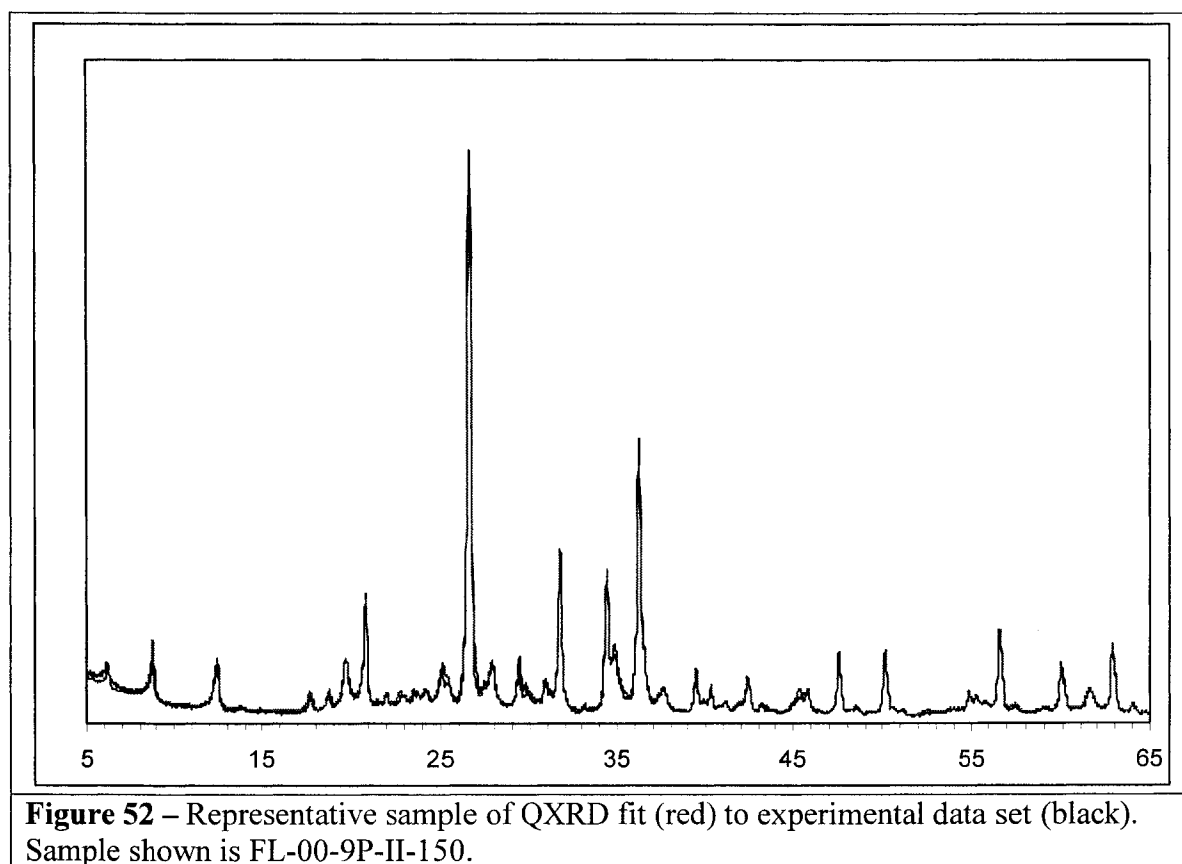
Figure 51 – Mineralogic data utilized to select SEM samples from core 9P. Lines show location of samples and measured mineralogy data. Note: top sample (I-2) does not have direct mineralogic measurements; line values on figure are interpolations. Hatched boxes show location of tephras.

between 2 to 4 times and determine the average %difference for each mineral phase. Total uncertainty reported for mineral phases are based on the QXRD analyses and calculated as the %difference of replicate analyses plus 2 standard deviations of the %difference. Table 9 shows the average uncertainty for each mineral phase and the totals, and includes the average %composition for the 20 replicate samples. In this study I found total uncertainty to range from 5.4% to 249.7% and the average %difference to range from 2.3% to 124.5% for the individual mineral phases. Uncertainty for the average total QXRD analysis was 7.0% and 2.5% for the %difference of the replicates (Table 9 and Figure 52).

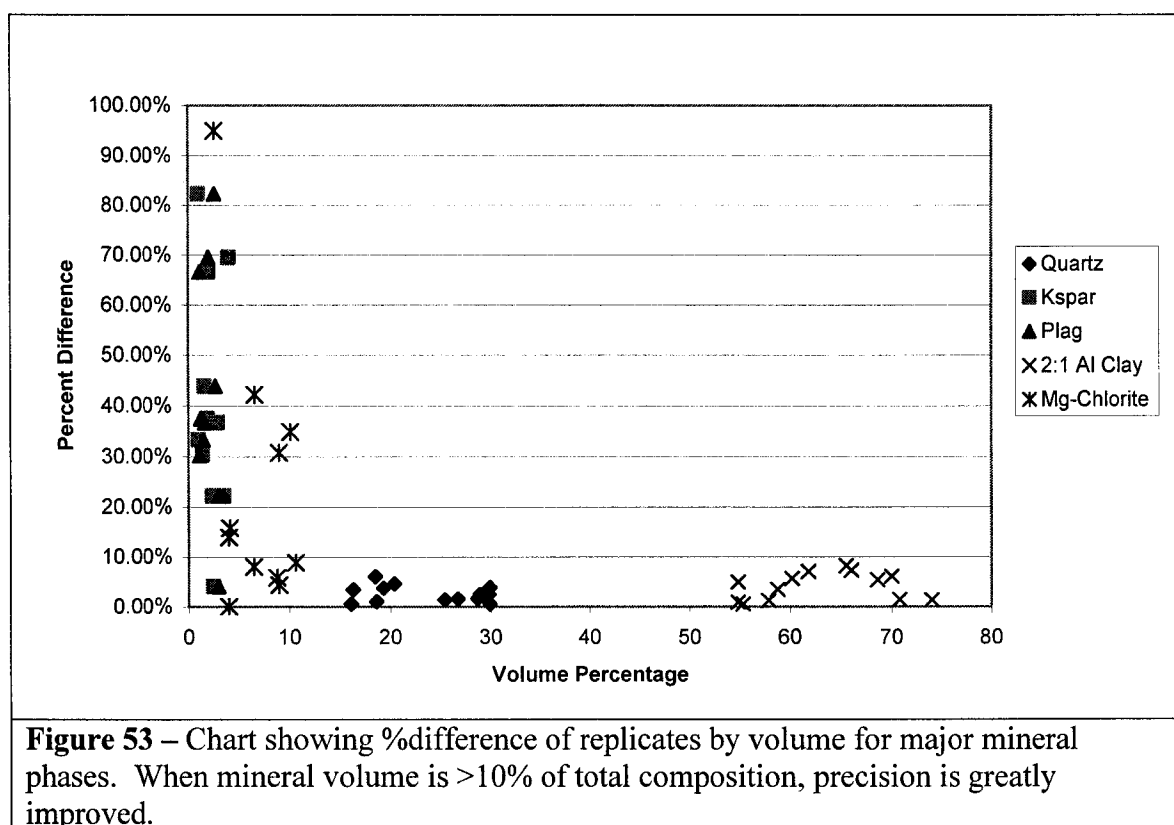
Mineral	Avg. %Composition	Avg. %Difference	2 St. Dev.	Total Uncertainty
Quartz	25.2	2.3%	3.0%	5.4%
Potassium Feldspar	2.4	40.4%	86.0%	126.4%
Plagioclase	3.5	16.3%	31.1%	47.5%
Calcite	1.2	34.0%	36.4%	70.4%
Mg-Calcite	0.2	67.1%	100.8%	167.9%
Dolomite	1.3	28.8%	49.9%	78.8%
Pyrite	0.3	48.7%	62.1%	110.8%
Siderite	0.1	74.0%	110.4%	184.4%
Opal	0.3	124.5%	119.2%	243.7%
Fe (oxy-)hydroxide	0.7	30.7%	55.3%	86.0%
Sum non-Clay	34.9	4.5%	9.9%	14.4%
Kaolin	0.8	66.2%	101.2%	167.4%
2:1 Al Clay	57.2	6.1%	14.2%	20.2%
Fe-Chlorite	0.9	77.4%	172.3%	249.7%
Mg-Chlorite	7.2	53.1%	115.1%	168.2%
Sum Clay	65.9	4.1%	8.0%	12.1%
Total Minerals	100.9	2.5%	4.5%	7.0%

Table 9 – Uncertainty for QXRD analyses from core 9P.

Analyzing the uncertainty data I found that percent composition of a mineral directly influences the resulting uncertainty. For example, when %composition is >10%, uncertainty is 11.8%, however when the mineral phase or total composition was < 10% average uncertainty jumped to 114.8% (Fig. 53). The average %difference for the two mineral phases with >10% composition (Quartz & 2:1 Al Clay) is 4.2%. This is double the <2% precision reported by Eberl (2003), but considering the differences in compositional complexity, may be comparable. Other studies using the same methodology with naturally occurring sediments either fail to report uncertainty (Kile and Eberl 2003) or only briefly mention the ~2%wt precision reported by Eberl (2003) (Eberl 2004; Hochella et al. 2005; Hein et al. 2006).

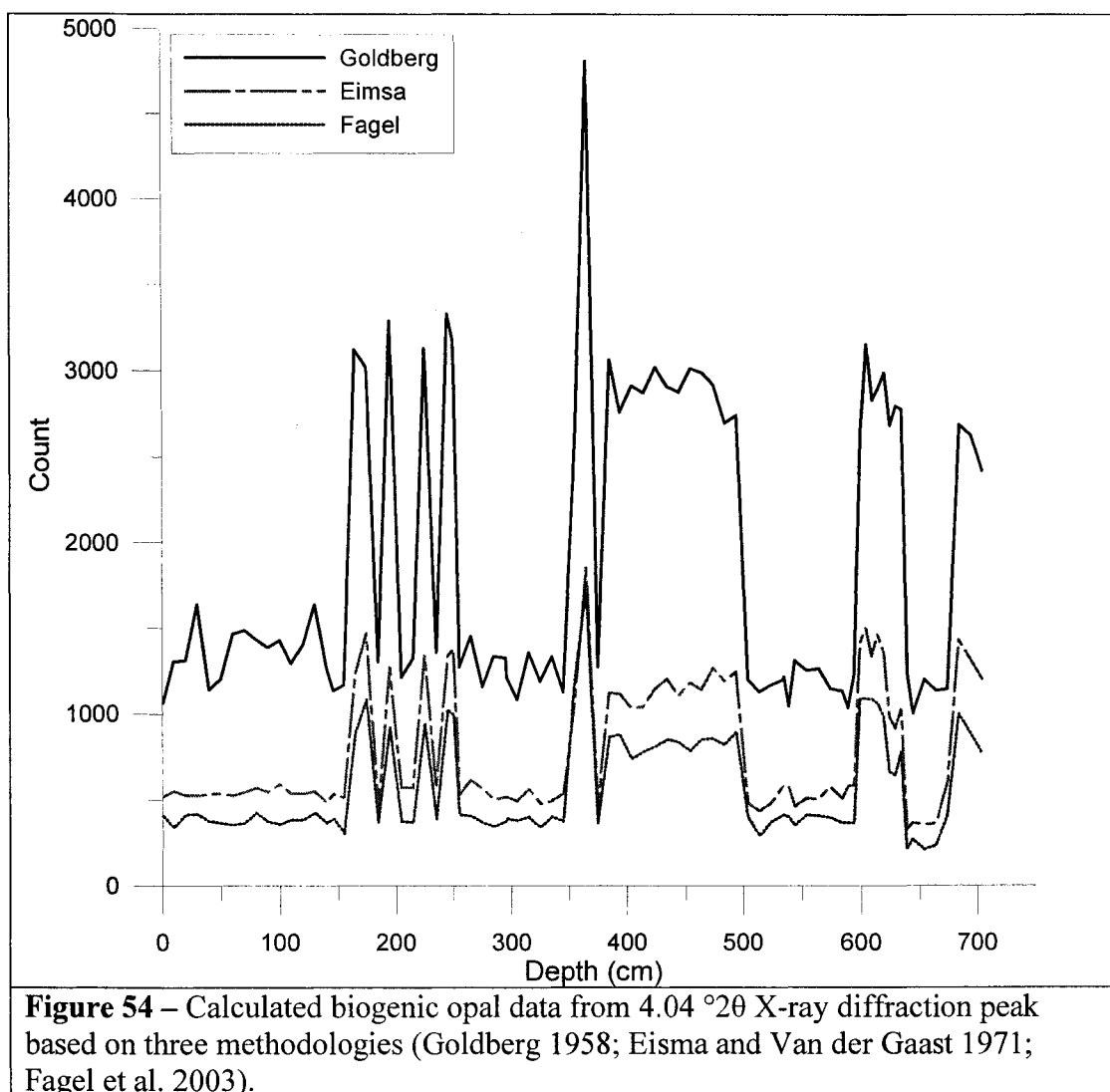


Our data suggests the QXRD methodology (Środoń et al. 2001; Eberl 2003) provides reasonable accuracy for mineral phases with %weight in excess of 10%. Data below this level likely suffer from resolution limitations and have correspondingly high uncertainties. However, given high enough variability these data may still provide significant paleoclimate information.



Two other types of mineralogic data were considered in this study in addition to mineral volume percentages; mineral ratios and calculated indices (i.e., kaolin/illite and illite crystallinity index). Uncertainty for mineral ratios simply is reported as the product of

the individual total uncertainties. The uncertainty of calculated indices proved to be somewhat problematic, as they are based on a graphical measurement of 2θ peak heights or widths. However, three independent measures of biogenic opal were made based on similar methodology (Goldberg 1958; Eisma and Van der Gaast 1971; Fagel et al. 2003). The three methodologies returned data sets with correlations that ranged from 0.95 to 0.97, although the magnitudes of the data varied (Fig. 54). Therefore, to utilize the three



methods as replicate measurements, they had to be normalized. Normalization was accomplished by calculating Z-scores in SPSS. Uncertainty for biogenic opal was then calculated as the %difference of Z-score for the individual value. To provide some measure of uncertainty for the illite crystallinity index, where no replicates are available, I show the QXRD illite uncertainty of 20.2%, which is partly responsible for variation in peak height and width.

Results and Discussion - In core 9P the illite crystallinity index lingers just below the transition line (0.38) between fresh and moderately weathered clay (Horiuchi et al. 2000), suggesting low chemical weathering. However, most of the variation in the crystallinity index is within the uncertainty. A few intervals of moderately crystalline illite exist in core 9P between 625-540, 510-470, 330-300, and 220-210 cm (Fig. 55). The most intense weathering illite occurs at 330cm, after an interval of relatively fresh illite. This is the only illite crystallinity peak that exceeds the uncertainty.

Excluding the Mazama tephra, the only %illite trends that exceed uncertainty are below 640 cm, the interval associated with the upward-fining beds (Fig. 54). The variability in %kaolinite and kaolinite/illite ratio is within the uncertainty over the entire length of the core. This is partly due to the low %kaolinite in the core contributing to the high uncertainty (see above). However, compared to the lower half of the core, the percent kaolinite and the kaolinite/illite ratio are generally elevated between 380-0 cm (Fig. 55). Although the trend that occurs within the uncertainty suggests higher weathering rates in

the upper part of the core, this trend is not supported by a similar trend in the illite crystallinity (Fig. 55).

Biogenic silica data has been utilized as an indicator of primary productivity, because diatom tests have been found to be the main source of biogenic Si (Bareille et al. 1990; Qiu et al. 1993; Bareille et al. 1998; Brauer et al. 1999; Talbot and Lærdal 2000; Fagel et al. 2003; Fedotov et al. 2004). The biogenic Si data in core 9P suggests intervals of greater productivity between 710-684, 638-599, and 494-385 cm, and oscillating productivity between 250-165 cm. These peaks and trends are generally outside uncertainty although some of the data points within the trends do not exceed the uncertainty. Variations in uncertainty within the data set result from the fact that uncertainty calculations were discretely applied to each point versus calculated as an average of all the points (see above). Transitions between trends are relatively abrupt, suggesting long periods of greater or lower productivity within the lake (Fig. 54 & 55).

Despite the suggestion of variations in biogenic Si in core 9P and the possibility that these reflect significant changes in lake productivity, qualitative visual examinations of core sediments do not support these interpretations. Randomly selected smear slides with high and low biogenic Si values and the previously mentioned SEM scans did not show high concentrations of diatoms where elevated XRD based biogenic Si suggested they should be present. Smear slides from the top of core 9P contained abundant diatom fragments, while biogenic Si suggests low productivity. Similar relationships were observed in other smear slides. The SEM scans also suggested the presence of very few

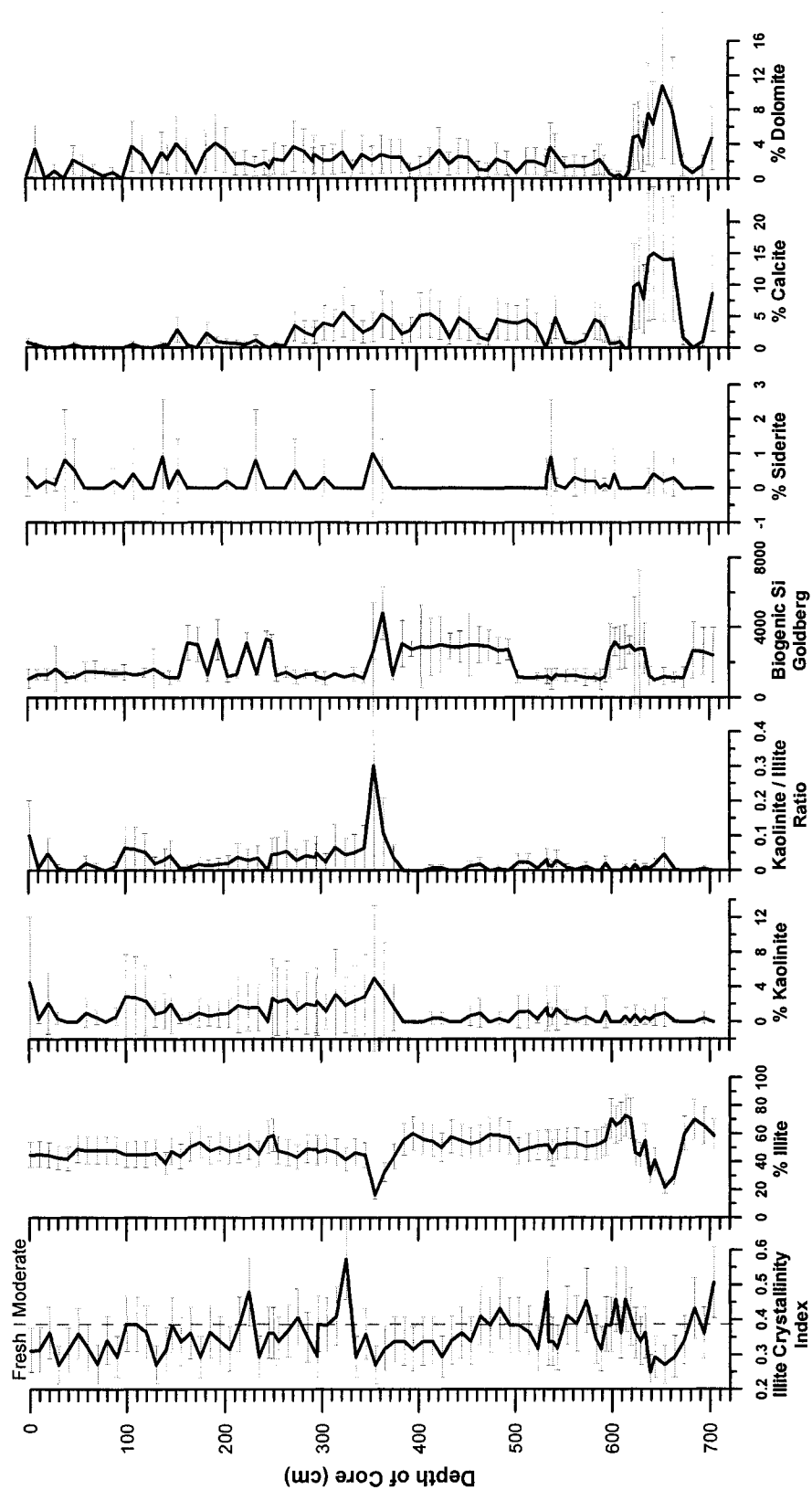


Figure 55 – Selected mineralogical data for core 9P with potential paleoclimate sensitivity. Mineralogical data based on 82 QXRD measurements. See Appendix E for graphs of complete mineralogical data set.

diatom frustules in samples with high biogenic Si. The SEM samples with the greatest abundance of diatoms were FL-03-16K-II-130 and FL-03-16K-II-129, located just above the Mazama tephra, where low biogenic Si values are present in core 9P (Appendix E, plates 1 & 2).

The variation of the %siderite is completely within the uncertainty and thus of little value for interpreting paleoclimate. As mentioned above, this is mostly due to the lower resolution of the QXRD analysis when mineralogic concentrations are < 10% (Fig. 53, Table 9). Carbonate mineralogy (%calcite and %dolomite) contain a number of peaks and trends that are outside the uncertainty and might provide some paleoclimate information. Variation in %calcite associated with the upward-fining beds below 640 cm is beyond the uncertainty although %dolomite, which shows the same variation, is overlapped by the uncertainty (Fig. 55). The trend of lowered %calcite values from 265 cm to the top of the core is outside the uncertainty, as are two peaks within this interval at 185 and 165 cm (Fig 55). The trend of %dolomite above 265 cm does not covary with the decreasing %calcite trend, although the magnitude of dolomite variability in this interval is greater. Dolomite is generally within analytic uncertainty above 265 cm, with exceptions at 50 and 10 cm where %dolomite peaks exceed the uncertainty. Visual inspection using the SEM did not reveal the presence of any authigenic carbonate grains (Appendix E, Plates 3 & 4), although EDS elemental maps for some of the SEM samples identified intergranular calcium concentrations suggestive of possible carbonate cement formation (Appendix E).

The overall resolution of mineralogic proxies in core 9P is limited. Data below 640 cm for carbonates, illite crystallinity, and %illite suggest increased hydrologic activity with low weathering indices and high carbonate input, which is in agreement with carbon/nitrogen and grain size data. The lower values of %calcite above 265 cm correlate with a lower trend in TIC, and the reductions above ~100 cm in %dolomite, kaolinite/illite, and %kaolinite correlate with reductions in TC and larger decline in TIC (see below). However, most of these correlations are tenuous since many data sets are within the uncertainty.

Carbon / Nitrogen

Background - Variations in the organic carbon/nitrogen ratio (C/N) have provided insight into the sources of carbon in lacustrine systems (Qiu et al. 1993; Jellison et al. 1996). C/N ratios below 10 indicate that organic carbon is largely from algal/phytoplankton sources. C/N ratios above 20 indicate dominance of carbon derived from vascular, cellulose-bearing terrestrial plants (Meyers and Lallier-Verges 1999). C/N values between 10 and 20 generally indicate a mixed source of algal and terrestrial plant material.

Higher C/N ratios from lake sediments have been used as indicators of greater terrestrial input associated with increased precipitation-derived runoff (Horiuchi et al. 2000; Wagner et al. 2000; McFadden et al. 2004). However, Meyers and Lallier-Verges (1999) caution that wetter climate intervals also can increase algal production in the absence of

terrestrial input, resulting in lower C/N ratios and an increase in total organic carbon (TOC) values. Furthermore, Kaushal and Binford (1999) found significant increases in C/N ratios that could only be attributed to deforestation and not to precipitation.

Research has suggested that C/N values shift lower due to algal production in nitrogen starved environments (Healey and Hendzel 1980; Hecky et al. 1993; Talbot and Lærdal 2000). Hecky et al. (1993) suggested that, in nitrogen-limited environments, C/N ratios < 8.3 indicate dominance by phytoplankton, whereas C/N ratios between 8.3 and 14.6 indicate mixed contributions. A terrestrial source of carbon from higher plants (C₃) is suggested by C/N ratios > 14.6 (Hecky et al. 1993; Horiuchi et al. 2000; Talbot and Lærdal 2000).

Changes in sediment TOC levels have been used as a proxy for primary productivity in lacustrine and marine environments (Qiu et al. 1993; Jellison et al. 1996; Johnson et al. 1998; Brauer et al. 1999; Li et al. 2000; McFadden et al. 2004). Positive correlations between TOC and productivity also have been demonstrated for carbon-poor oligotrophic lake systems (Cohn 2003). When examined in conjunction with C/N values, TOC can be used to determine if primary productivity is largely aquatic (C/N < 8.3) or terrestrial (C/N > 20) (Finney and Johnson 1991).

Methods – Total carbon (TC) and nitrogen (TN) data were obtained at 5 cm intervals in core FL-00-9P to a depth of 610 cm. Below 610 cm, data were obtained at 1 cm intervals to improve resolution at the postglacial transition. Total carbon and nitrogen analyses

were performed on dried samples by gas chromatography on a CHNS-1110 elemental analyzer (EA). Total inorganic carbon (TIC) analysis was performed on a Coulometrics Carbon Analyzer on the same core intervals as the total carbon and nitrogen measurements. Total organic carbon (TOC) was determined indirectly by differencing the total carbon and inorganic carbon values. The C/N ratio is calculated as a molar ratio of TOC and TN. All carbon and nitrogen results in this study are reported as percent by weight of dry sample.

Uncertainty - Accuracy for all analyses was determined by performing duplicate measurements on every 10th sample. Standard errors were calculated from mean percent differences of the duplicate measurements and the associated standard deviations. Equipment precision is reported at the 1σ confidence level. Precision testing of the CHNS-1110 analyzer was based on a laboratory reference soil standard, ThermoQuest SpA. P/N 338 40025, which was measured after calibration and every 10th sample. For total carbon, the percent difference between the mean of the measured values and the standard was 1.1%. The relative standard error (std. error/mean) was 0.1%. Precision testing of the coulometer was based on a calcium carbonate reference standard measured 3 times before the first sample run and following every 10th sample. The percent difference between the mean of the measured values and the standard was 0.33%, the relative standard error was 0.27%.

Uncertainty analyses in this study are reported as %difference of replicate samples, plus 2 standard deviations of the %difference. A total of 19 replicate measurements were

recorded on the elemental analyzer for core 9P. The uncertainty for total carbon was 13.3% with an average %difference of 2.8% (Table 10). Uncertainty for the total nitrogen data was considerably higher at 171.6%, with the average %difference at 35.6%. Nitrogen values in Flathead Lake are very low (<0.12%) and in some intervals were below the EA detection limits. The low TN values are the leading cause of the high uncertainty figures. Coulometer TIC uncertainty is based on 21 replicate samples from core 9P with total uncertainty, amounting to 11.8% with an average %difference of 2.6% (Table 10). Total organic carbon is a calculated value and therefore the uncertainty is calculated from the components, as the TIC uncertainty plus the TC uncertainty. Uncertainty for the C/N ratio was calculated as the sum of the TC and TN uncertainties (Table 10). The C/N uncertainties also are uncommonly high due to low TN values.

	TN	TC	TIC	TOC	C/N
Avg. %difference	35.6%	2.8%	2.6%		
2 St. Dev.	136.0%	10.5%	9.3%		
Total Uncertainty	171.6%	13.3%	11.8%	25.1%	184.9%
Table 10 – Uncertainty values for carbon / nitrogen data					

Results and Discussion – Overall Flathead Lake sediments contain very low levels of total carbon, generally <1.8% (Fig. 56). However, from the bottom of the core to 622 cm, total carbon (TC) contains two sequences of peaks in which TC reaches a maximum of 3.8%. These peaks in TC are associated with upward-fining beds identified in the median grain size record (Figs. 43 and 44). Peak values exceed the uncertainty range of the baseline TC values between the two sequences and above the uppermost upward-

fining bed. At 621 cm TC decreases to $< 0.6\%$, then gradually increases between 622 and 528 cm to $\sim 1\%$. Between 524 and 150 cm TC values fluctuate between 0.8% and 1.8% mostly within the uncertainty, excluding the Mount Mazama tephra. At 150 cm TC decreases outside of the uncertainty to $< 1\%$ and remains at that level to the top of the core.

TC tracks TIC from the bottom of the core to 185 cm (Fig. 56). In this part of the core TIC typically represents more than 50% of the TC, although it exceeds 90% in the upward-fining beds (622-670 and 692-705 cm). Above 180 cm TIC contribution is $< 50\%$, except a peak at 155 cm and three small peaks at 115, 50, and 10 cm, where TIC reaches $\sim 50\%$. At 105 cm TIC drops sharply, outside the uncertainty, and remains at the lowest levels to the top of the core (Fig 56).

Total organic carbon (TOC) in Flathead Lake is generally low ($< 1\%$), increasing from a low ($< 0.1\%$) at 622 cm to the top of the core, but the trend remains mostly within the uncertainty, excluding the tephra (Fig. 56). A few narrow peaks in TOC occur between the bottom of the core and 633 cm and are associated with some of the upward-fining beds. Relatively higher TOC levels exist between 540-525 cm (up to 0.57%). Above 525cm, TOC increases upward through the rest of the core, excluding the Mount Mazama tephra. However, nearly all of this upward increase in TOC is within the analytical uncertainty of the data (Fig. 56).

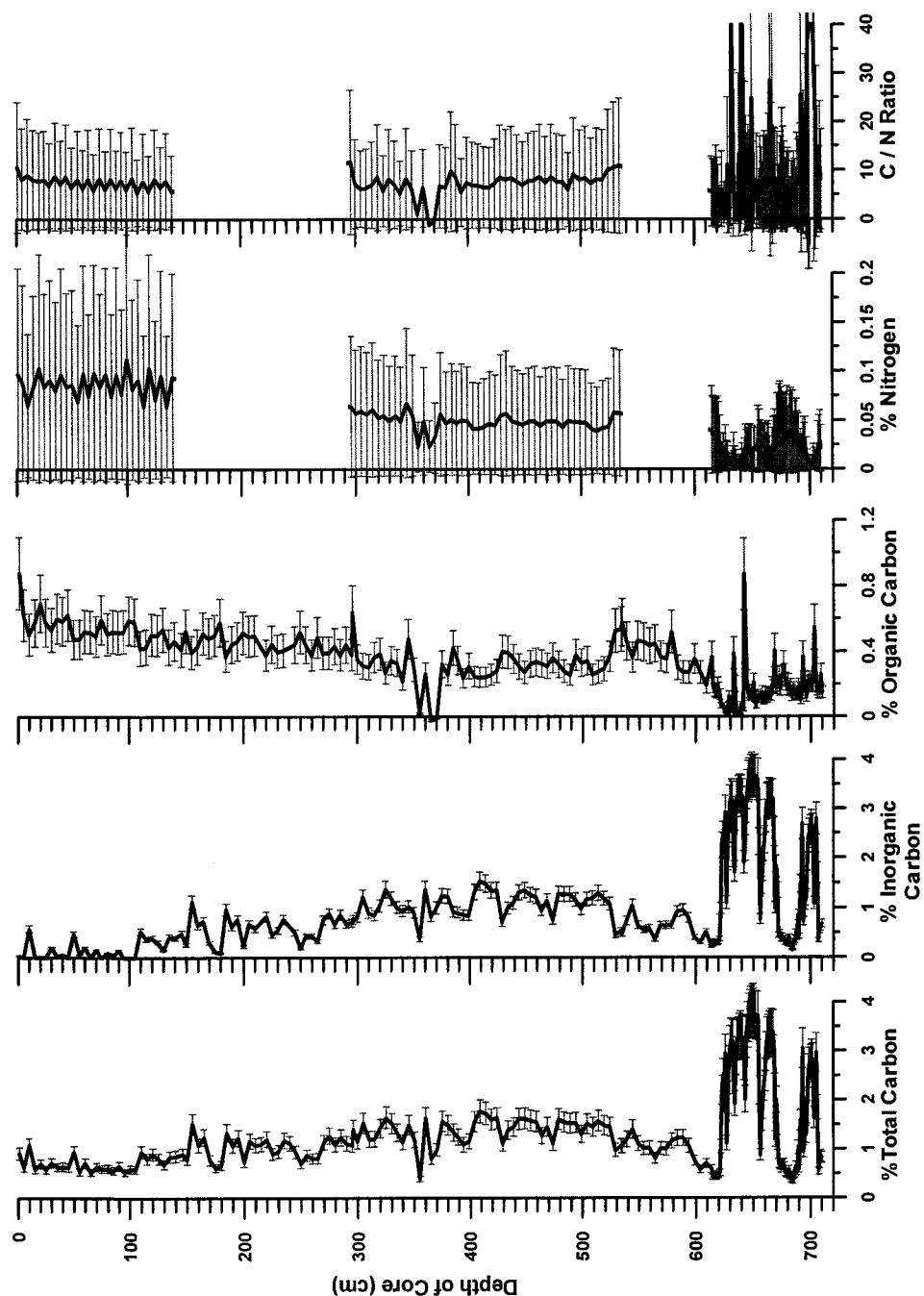


Figure 56 – Carbon and nitrogen data for Flathead Lake core 9P. Nitrogen data for intervals of 614-535 and 295-145 cm are below detection limits of the EA, C/N ratio and %Nitrogen could not be calculated for these intervals. Some error bars and a couple peaks truncated on lowest section of C/N graph.

Total nitrogen in core 9P is very low ($< 0.1\%$) and was below detection limit of the EA between 614-535 cm and from 295-145 cm (Fig. 56). The lack of nitrogen data created two large gaps in the nitrogen and C/N curves, limiting their value for paleoclimate interpretations. The low TN values also contribute to the high TN and C/N uncertainty. Consequently all but a few C/N ratios at the bottom of the core are within the calculated uncertainty. Where available, C/N ratios are mostly below 10 (Fig. 56) indicating lake primary productivity from algal plants. Values > 20 are present only as narrow peaks associated with the upward-fining beds.

The low TN values indicate that Flathead Lake has remained a nitrogen-starved system for the duration of available core 9P data. I also examined the C/N data based on the ratio breaks suggested by Hecky et al. (1993) for nitrogen starved systems (Fig. 57). Comparing the two methods, only 1 additional data point (from a former total of 12) crosses the terrestrial boundary (14.6) for low N system, with the bulk of the data remaining < 8.3 and suggestive of an organic carbon system dominated by algal production. All but one of the C/N data points in excess of 14.6 are directly associated with the upward-fining beds (labeled 'turbidite' in Fig. 57). The remaining point (labeled 'lacustrine' in Fig. 57) is located between two upward-fining beds. It is likely that this sample received some detrital organic material from the surrounding flood beds.

In general, variability in the carbon and nitrogen data is small and within the uncertainty, thus limiting the ability to construct paleoclimatic interpretations. The low nitrogen values are similar to current levels in Flathead Lake (Spencer and Ellis 1990) suggesting

oligotrophic lake conditions since glacial retreat, ~13,000 cal. yr. BP. The intervals where TN values are below detection suggest that lake productivity exceeded external TN input (Wetzel 1983). A trend of increasing values up core in TOC and TN imply higher primary productivity in the lake across the Holocene, although these trends are within the analytic uncertainty (Fig. 56).

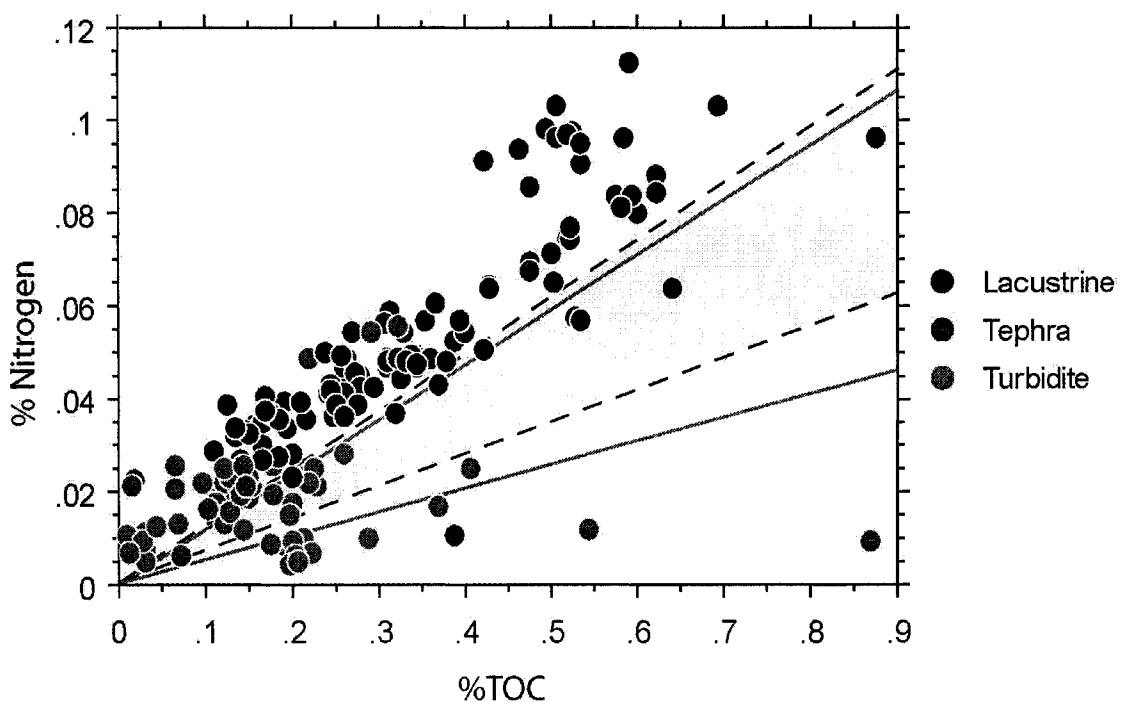


Figure 57 – Scatter plot of %N vs. %TOC for core 9P sediments showing that most of the organic material in Flathead Lake is derived from within the lake by algal production (area above upper dashed line). Dashed boundaries are based on Hecky et al.(1993) for nitrogen starved systems (upper line = C/N of 8.3, lower = 14.6), solid blue lines are boundaries (upper = 10, lower = 20) based on Meyers and Lallier- Verges (1999). Yellow fill area shows value range of mixed algal/terrestrial organic source material. All but one point of terrestrially derived organics (below lower dashed line) is associated with the upward-fining beds (turbidites) from the lowest part of the core (see text).

In core 9P, carbon sources inferred from C/N data mostly indicate an algal-based carbon system, although some parts of the core show a mix of terrestrial and algal input. Late glacial upward-fining beds, interpreted to be flood deposits related to deglaciation, commonly show substantial terrestrial carbon input, although this may in part be function of extremely low levels of lake productivity during deglaciation. With this said, C/N ratios have very limited variability in the post-glacial period, suggesting an algal based oligotrophic lake since deglaciation.

In Lake Biwa, Japan, periods of increased C/N ratios and TOC that are coeval with changes in the pollen record that are inferred to record productivity and temperature increases (Meyers et al. 1993). In Flathead Lake this relationship also may be present, represented by an increasing C/N and TOC trend in the upper part of the core (Fig. 56), although both of these trends are within the uncertainty. However, caution must be used with TOC data in oxic bottom waters, such as Flathead Lake, where post-burial degradation may decrease organic carbon levels until burial in anoxic sediments (Meyers and Teranes 2001).

In the lowest part of the core the upward-fining beds contain high TIC and C/N values, suggesting terrestrial input into the lake. The coarse grain size of these beds (Fig. 43) supports an influx of detrital carbonate and organic material at this time. Qualitative SEM examinations of the core sediments suggest that all carbonate material in the upward-fining beds is detrital (Appendix F). Additionally, the SEM examination failed

to observe any authigenic carbonate in the samples from cores 9P or 16K. These results suggest that the TIC data set is primarily detrital and not an indicator of lake productivity.

Conclusions:

Paleoclimate reconstructions are accomplished by analysis of proxy data sets, which are inferred to represent one or more climate conditions that cannot be directly measured.

Since most proxies are a partial function of multiple climatic inputs, proxies are best evaluated in groups for the most accurate reconstruction of the preserved climatic signal.

However, multiple proxies can behave out of phase for all or a portion of the record.

Discrepancies between proxy signals can be caused by a number of factors that include:

1) varying sensitivity of the proxies to climate change; 2) sensitivity of a proxy to multiple climatic conditions (i.e., dependence on both temperature and precipitation); 3) lag effects where a proxy responds to a climatic change after a period of time (i.e., the time taken by a tree to mature before releasing pollen); 4) dilution of a proxy signal due to destructively interfering multiple signals within a large montane watershed; 5) shifts or missing peaks in a proxy due to different analytical resolution; 6) differential fractionation of sediments across the lake bottom due to currents and other unquantified depositional processes; and 7) other poorly understood or unquantified factors, such as proxy preservation and post-depositional reactions. Additionally it should be mentioned that most proxies only reflect qualitative changes in climate conditions and do not provide a quantitative estimate of the magnitude of the climate change. This is because the magnitude of a proxy peak typically does not reflect a known absolute value that directly correlates to the measured climate change.

A certain amount of uncertainty exists in all measurements. This uncertainty must be considered to fully evaluate the utility of the proxy for paleoclimate interpretation. Unfortunately, most paleoclimatic reconstructions fail to fully consider uncertainty in their interpretations or only provide it a passing mention (e.g., Harrison and Digerfeldt 1993; Brauer et al. 1999; Horiuchi et al. 2000; Talbot and Lærdal 2000). In contrast, many paleoclimate studies recognize chronological uncertainty (e.g., Mayle and Cwynar 1995; Yu 2000; Thamban et al. 2002).

In this study uncertainty calculations were conservative and sought to provide climate interpretations based on > 95% probability. Using this approach, many of the small variations in the Flathead Lake record are less than convincing with respect to the proxy connection to a climate change.

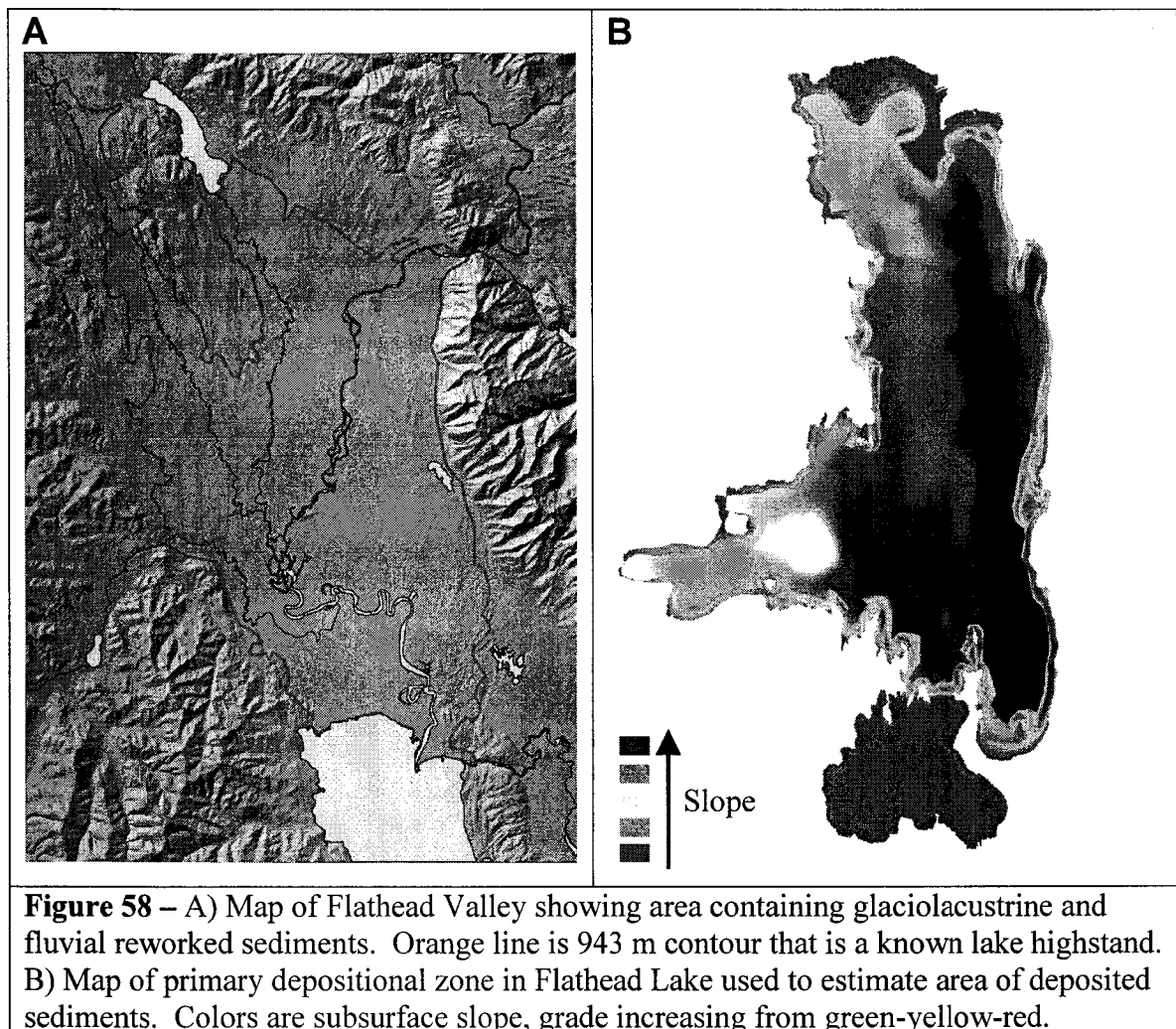
Interestingly, proxy variations of a magnitude similar to those found in the Flathead record have been deemed climatically significant in other published studies (i.e., Qiu et al. 1993; Yuretich et al. 1999; Talbot and Lærdal 2000). The comparison of multiple proxies is becoming standard practice when using lacustrine sediment records to infer a climate change. This approach is particularly important when proxy variability is within the uncertainty (i.e., Mayle and Cwynar 1995; Schmidt et al. 2002; Andrews and Giraudeau 2003; Stevens et al. 2006).

An additional buffer that needs to be considered in the interpretation of the Flathead Lake Holocene paleoclimate record is the effect that reworked glaciolacustrine sediment stored in the Flathead Valley may have had on the paleoclimate signal. Glaciolacustrine varved sediments have been mapped throughout the Flathead Valley (Smith 2004) and are generally confined to within the 943 m elevation contour line (Fig. 58A).

Glaciolacustrine outcrops in the Kalispell area have exposures up to 19 m in height and show indications of being eroded by the upper Flathead River. The age of these sediments is poorly constrained, although Smith (2004) differentiated between late Pleistocene and Holocene deposits based on color and morphology. The older beds are attributed to a glacial lake stage (Smith 2004) based on their similarity to varved deposits in the Mission Valley attributed to glacial Lake Missoula (Levish 1997).

To calculate the potential glaciolacustrine sediment erosion due to downcutting and mobilization, I used various mean elevations of lake deposits in the Flathead Valley. In ARCGIS v.9 I could then calculate the volume of sediment from the sediment terrace down to the current topography. Estimates of eroded glaciolacustrine deposits range from 3.75 to 22 km³. However, an assessment of the volume of sediments that may have been eroded due to wave action of lake stands above current lake elevation is unconstrained. These reworked sediments would likely have been redeposition locally as lacustrine sediments in the broad plain above Flathead Lake. Estimates based on well logs suggest that the clay and silt layers, interpreted as lacustrine deposits, between Flathead Lake and Kalispell are as thick as 150 m with some interbedded gravels (Smith 2004).

Within Flathead Lake the coarse upward-fining beds are posited to be flood deposits that represent a period of glacial retreat from the Flathead Valley. The Glacier Peak tephra ($13,180 \pm 120$ cal. yr. BP) is located above these beds in the lake cores and also is found in Flathead Valley where it is overlain by eolian sediments. These observations support the interpretation that the Flathead Valley had undergone glacial retreat and drying by this time (Smith 2004). Thus, I could calculate the volume of sediment deposited in the



lake, since the glacial retreat, based on the sediment thickness of Flathead Lake cores between the sediment water interface and the upward-fining beds. These calculations are based on the major depositional regions within the lake (Fig. 58B) and consider coarsening of sediments closer to the river mouth. I estimate sediment deposition in Flathead Lake since glacial retreat (i.e., above the last upward-fining bed) to range from 1.9 to 3 km³.

Whereas the actual reworked glaciolacustrine percent contribution to the core 9P sediments is unquantified, based on these crude sediment erosion and deposition calculations it is likely to be substantial. Particularly when the composition of the glaciolacustrine sediments is considered, these very-fine grained sediments are most likely to be transported to the middle of the lake and therefore likely contributed to core 9P sedimentation.

I propose that erosional reworking of previously deposited fine-grained glaciolacustrine sediments in the upper Flathead valley buffered the paleoclimate signals derived from time-series proxy data and, at times, provided the main signal captured by the climate proxies. This finding suggests that caution must be exercised in using proxies records developed from high latitude oligotrophic lakes that derive most of their climate signal from detrital sources. Many of these lakes were larger during post-glacial periods, and many have glaciolacustrine sediments mantling large portions of their watersheds.

Climatic Inferences:

The sedimentary record recovered in core 9P does not reach the Last Glacial Maximum (LGM) (Levish 1997; Smith 2004) as indicated by the chronology (Chapter 2) and by the lack of the progressively thickening (down core) varved record observed in longer cores from Flathead Lake (Hofmann et al. 2003). The lack of glacial varve sedimentation suggests that the lake was not seasonally ice covered at the time of deposition of the lowermost beds in core 9P (Anderson 1993). The lower most part of the core contains a series of upward-fining beds that have been identified in subsequent cores throughout the southern part of Flathead Lake (Hofmann et al. 2003). High C/N ratios of 25-40 indicate vascular terrestrial input of carbon (Meyers and Lallier-Verges 1999) and suggest that the relatively coarse-grained sediment flows were derived from outside the lake (Figs. 44 and 56). The lower series of upward-fining beds passes upcore into a dm-thick section of very fine-grained laminated sediments that are not varved.

The upward-fining beds and the interbedded very fine-grained more homogeneous sediments below the Glacier Peak tephra are temporally correlative with the late stages of the Bolling/Allerod episode (BA) reported in the GRIP cores (Asioli et al. 2001) and elsewhere (e.g., Alley et al. 2002; Hendy et al. 2002). The Bolling/Allerod is described as a rapid warming as recorded in $\delta^{18}\text{O}$ proxy and the time of postglacial sea level rise (Yu 2000; Alley et al. 2002; Seltzer et al. 2002; Ciampo 2003). Paleoclimate models suggest that the Bolling/Allerod episode of warm and relatively wet climate rapidly ended, giving way to a cool/dry glacial climate around 13,000 cal. yr. BP (Walker 2001). This cool/dry period, called the Younger Dryas (YD), is identified in climate

reconstruction records as a rapid climate reversal to cold conditions that lasted for between 1,000 and 1,500 years (Alley 2000; Yu and Wright 2001; Kovanen and Easterbrook 2002). The YD is correlative with the last Bond cycle and H0 Heinrich layer (Bond and Lotti 1995; Alley 1998; Yu and Wright 2001). However, paleoclimate reconstructions differ on the question as to whether the YD was a wet or dry period (Renssen et al. 2000; Lambeck et al. 2002; Renssen and Vandenberghe 2003).

In Flathead Lake no physical evidence (such as increase in grain size or varved sediments) exists to suggest glacial ice returning to the valley, and other proxies remain within uncertainty. It is likely that the YD ice advances documented just north in British Columbia (Mathewes et al. 1993; Mayle and Cwynar 1995) either did not cross the Libby Divide into the Flathead watershed or that glacial sediments were trapped in smaller upstream lakes, minimizing any signal in Flathead Lake sediments.

Researchers studying Holocene sediments in Canada and the Great Plains have documented an anomalous warm interval commonly referred to as the mid-Holocene Hypsithermal (ca. 4,700 to 9,000 yr. BP) (Mathewes and Rouse 1975; Alley 1976). In Flathead Lake, few statistically significant shifts are recorded in the proxy data to support this warming. An exception to this generalization is significant changes in the biogenic Si time-series data, although this proxy is suspect based on SEM observations (see text above). Most time-series data sets for core FL-00-9P are within analytic uncertainty, rendering climatic interpretation based on these data alone tenuous at best. Two noteworthy trend changes occur at ~6,500 cal yr. BP when both %calcite and %TIC

decrease significantly, and at 4,500 yrs when the %TIC and %TC decrease again.

Although, SEM observations suggest that TIC and carbonate minerals are primarily detrital and poorly connected as a climatic proxy, suggesting a non-climatic control.

My examination of the historical climate records for the watershed suggests caution must be exercised when using lake records to reconstruct paleoclimate in mountainous topography. Mountains that separate valley lakes serve to partition the direction and magnitude of climate change in complicated topography. In small lake systems, these variations may provide a local signal that is only partially in phase with the more regional paleoclimate picture. Large drainage systems can integrate these climatic variations, although the integration also serves to dampen proxy signals recorded in the sediments. Reworking of older lake sediments within the basin represents another possible source of noise or buffering in the paleoclimate signal. Lastly, uncertainty in the time-series data sets must be considered to accurately evaluate data set variations and best improve the quality of paleoclimatic interpretations derived from lake sediments.

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Appendix A
Radiometric Dating and Tephra Geochemistry



January 12, 2001

Professor Marc S. Hendrix
Department of Geology
University of Montana
Missoula, MT 59812-1019

Dear Marc,

I have enclosed the results on the samples from Flathead Lake, unused tephra, and the microprobe mount (under separate cover). There are no surprises in the results---they are what you expected.

The composition of the glass in FL-00-9P-III 45-46 is quite consistent and is an excellent match (similarity coefficient = 0.99) to that in a sample collected at Llao Rock at Crater Lake. Thus 45-46 represents either the climactic eruption of 6850 BP or a precursor approximately a few hundred years earlier. My educated guess is that your sample is mostly likely from the climactic eruption.

Sample FL-00-9P-II 78-79 consists mostly of Mazama tephra with a few shards of a more mafic glass of unknown origin. The bulk composition is a good match (similarity coefficient = 0.97) to published data (Westgate et al., 1970) on a sample of Mazama tephra and when the composition of the more mafic component is removed (see FL-00-9P-II 78-79, glass 2 in Table 1) the coefficient increases to 0.99. I don't trust the match of Mount St Helens T for glass 1 in this sample as the match for two of the more reliable element, Fe and K, is considerably off.

Sample FL-00-9P-V appears to be from one of the three major Glacier Peak eruptions (B,M, or G) which took place over a span of a few hundred years around 11,200 BP. Tephra from these eruptions cannot be distinguished on the basis of EMP determined glass compositions. The similarity coefficients of the matches range from 0.96 to data reported by Westgate et al. (1970) to 0.99 for a sample LIB-B I will soon publish on and am pretty sure is from Glacier Peak.

If you have any questions regarding these analyses please do not hesitate to call or email me (foit@mail.wsu.edu).

Sincerely,

Nick

Franklin F. (Nick) Foit, Jr.
Professor and Director of Microbeam Lab

Sample name: Hendrix FL-00-9P-V, 74-75												
SiO2	TiO2	Al2O3	MgO	CaO	BaO	MnO	Fe2O3	Na2O	K2O	Cl	Total	
77.31	0.21	12.80	0.24	1.22	0.00	0.03	1.09	3.52	3.42	0.16	100.00	
1.00	0.25	1.00	0.25	1.00	0.00	0.00	1.00	1.00	1.00	1.00	weighting factor (only for oxides in bold type)	
Similarity Coefficients for 15 closest matches												
SiO2	TiO2	Al2O3	MgO	CaO	Fe2O3	Na2O	K2O	Cl	Weighted avg	sim coef		
0.998	1.000	0.994	0.958	0.967	0.991	1.000	0.997	0.990	0.997	0.990	25-Aug-95 OR	77?
0.998	0.952	0.991	1.000	0.984	1.000	0.964	0.965	0.983	0.965	0.983	8-Jul-94 WA	BP
0.999	1.000	0.999	0.958	1.000	0.901	0.992	0.997	0.981	0.997	0.981	18-Oct-93 MT	GP Manyberries (G), 11200 BP ?
0.998	0.952	0.990	1.000	0.967	0.972	0.959	0.994	0.980	0.994	0.980	31-May-94 WA	BP
0.989	0.952	0.993	0.958	0.975	0.991	0.954	0.974	0.979	0.974	0.979	8-Jul-94 WA	BP
0.989	0.905	0.994	1.000	0.951	0.973	0.967	0.986	0.977	0.986	0.977	8-Jul-94 WA	BP
0.999	0.905	0.991	0.960	0.984	0.955	0.959	0.977	0.974	0.977	0.974	8-Jul-94 WA	BP
0.999	0.952	0.992	1.000	0.984	0.908	0.941	0.997	0.971	0.997	0.971	29-Dec-93 WA	BP
0.985	0.952	0.932	0.958	0.992	0.972	0.968	0.959	0.970	0.959	0.970	MT	Glacier Peak (G)?
0.982	1.000	0.920	1.000	0.967	0.963	0.992	0.956	0.966	0.956	0.966	MT	Glacier Peak (G)?
0.997	1.000	0.993	0.889	0.953	0.982	0.964	0.927	0.966	0.927	0.966	31-May-94 WA	BP
0.996	1.000	0.975	0.890	0.968	0.956	0.957	0.933	0.965	0.933	0.965	31-May-94 WA	BP
0.988	0.952	0.976	0.958	0.951	0.940	0.944	0.988	0.965	0.988	0.965	1-Mar-94 WA	BP
1.000	1.000	0.986	1.000	0.992	0.886	0.941	0.962	0.964	0.962	0.964	18-Oct-93 WA	GP Manyberries (G), 11200 BP ?
0.995	0.955	0.993	0.828	0.992	0.932	0.912	0.991	0.963	0.912	0.963	423 S	0 GP lower layer
number of records searched: 1145												

Notes

Sample LIB-B, west of Mt Hood, OR, Jim Chatters, Applied Paleoscience, 648 Saint Street, Richland, WA, 99352

Sample Gough #4, Lapilli below ash, Kuehne Locale, East Cove Profile, Sw1/4NW1/4SW1/4, Sec 11, T28N, R33E, WA, Stan Gough, Archaeological & Historical Services, Eastern Washington University, Cheney, WA

Sample from terrace/ash sediments location SE1/4NW1/4SEC 18, T20N, R21E, Sloan Mont, 7.5 Quad, MT, Dale Stradling, Dept of Geography-Anthropology, EWU

Sample 93-32 from Arch excavation site 45CH425, Osprey Camp Site on Wenatchee River terrace 3 m downstream from Lake Wenatchee, T27N, R17E, Sec 36, NW1/4, Plain, WA 7.5 Quad

Sample Gough #5 (Sample 12), Lower Spring Canyon, Sec 16, T28N, R31E, Grand Coulee Dam 7.5 Quad, Stan Gough, Archaeological & Historical Services, Eastern Washington University, Cheney, WA

Sample Gough #2, Upper Ash, Porcupine Bay, Lake Roosevelt, WA, Stan Gough, Archaeological & Historical Services, Eastern Washington University, Cheney, WA

Sample Gough #6, Beebe Pt, NE1/4SW1/4SE1/4, T27N, R23E, Chelan Falls 7.5 Quad, WA, Stan Gough, Archaeological & Historical Services, Eastern Washington University, Cheney, WA

Sample from Spring Canyon, Lake Roosevelt, WA, Dale Stradling, Dept. of Geography and Anthropology, Eastern Washington University

5.52-5.53m, Guardipee Lake, NW Mt. Bamnosky (1989), QR, 31, 57-73.

5.00-5.05m, Guardipee Lake, NW Mt. Bamnosky (1989), QR, 31, 57-73.

Sample 93-15 from Arch excavation site 45CH425, Osprey Camp Site on Wenatchee River terrace 3 m downstream from Lake Wenatchee, T27N, R17E, Sec 36, NW1/4, Plain, WA 7.5 Quad

Sample 93-26 from Arch excavation site 45CH425, Osprey Camp Site on Wenatchee River terrace 3 m downstream from Lake Wenatchee, T27N, R17E, Sec 36, NW 1/4, Plain, WA 7.5 Quad

Sample 1994, 1B, depth = 1.1m, surficial deposits in Yakima Canyon, southern Kittitas County, SE1/4NW1/4, Sec 28 T16N, R19E, B. Lenz, Central Washington University

Sample YRSPR3JF1-1 (upper ash), depth 1.1m, Yakima Ridge, Yakima Training Center, K. Boreen, Archaeological & Historical Services, EWU

Westgate, Smith and Tomlinson (1970) In Early Man & Environments in NW North America, p. 13-34, R.A. Smith & J.W. Smith eds.

weighted at

Tephra Glass Comparison

1/12/01

Sample name: Hendrix FL-00-9P-III, 45-46

SiO2	TiO2	Al2O3	MgO	CaO	BaO	MnO	Fe2O3	Na2O	K2O	Cl	Total
73.09	0.43	14.50	0.46	1.67	0.00	0.03	2.14	4.81	2.68	0.19	100.00
1.00	0.25	1.00	0.25	1.00	0.00	0.00	1.00	1.00	1.00	weighting factor (only for oxides in bold type)	

Similarity Coefficients for 15 closest matches

sim coef

SiO2	TiO2	Al2O3	MgO	CaO	Fe2O3	Na2O	K2O	Cl	wt% avg	rec#	Std	Date	State	Source/Access	Notes	weighted as
0.999	0.977	0.999	0.979	1.000	0.995	0.984	1.000	0.995	0.995	914	S	14-Apr-95	OR	Mazama Liao Rock 7000 BP	Cloud Cap pumice collected by Mehlinger, Wigand and Bacon on Aug 22, 1982 at Cloud	
0.997	0.977	0.995	0.939	0.971	1.000	1.000	0.996	0.990	0.990	346			OR	Mazama/6850	Willowdale	
0.997	0.977	0.994	0.978	0.982	0.977	0.998	1.000	0.990	0.990	1036		7-Mar-97	WA	Mazama 6850 BP ??	Sample from sediments within rock shelter on upper Wenatchee River above	
0.997	0.977	1.000	0.939	1.000	0.991	0.960	1.000	0.989	0.989	797		2-Sep-94	NV	Mazama Climatic 6850 BP ?	Leavenworth, WA (Winton 7.5 Quad, NW1/4, NW1/4, NE1/4 of Sec 21, T25N, R17E);	
0.998	0.977	0.999	0.979	1.000	0.986	0.954	0.996	0.988	0.988	1039		5-Mar-97	NV	Rock/Climatic 6850-7000 BP ??	Rebecca Stevens Archaeological & Historical Services, Eastern Washington University, Chanty, WA, 99004	
0.998	0.977	0.989	1.000	1.000	0.967	0.976	0.993	0.987	0.987	873		3-Jan-95	MT	Mazama Climatic 6850 BP ?	Sample 691-188; in soil horizon Bw1, depth 15-25cm bs. sand dune, Kootenai National Forest, Rebecca Timmons, Kootenai National Forest, 506 Hwy 2 West, Libby, MT 59923	
0.997	1.000	0.996	0.958	0.988	0.986	0.966	0.993	0.987	0.987	705		31-Jan-94	OR	Mazama 6850 BP	Sample 813-01-47-ASH recovered from Unit N196E174 at a depth of 30-35 cm from Site 35KL2101 (La Sere Site) in Drews Valley, roughly 17 miles west of Lakeview, OR, Dennis Jenkins, OR Museum of Anthropology, O of O	
1.000	0.930	0.999	1.000	0.964	1.000	0.976	0.969	0.986	0.986	188			ID	Mazama/6850	101H1639 Pittsburg Landing, B. Cochran, Pittsburg Arch. Project, USFS	
0.999	1.000	0.997	0.979	0.988	0.977	0.964	0.989	0.986	0.986	1102		3-Jan-97	WA	Mazama Climatic 6850 BP ??	Sample 45KT344 Tephra 2, depth 6.1m along Squaw Creek (SW1/4, SE1/4 of Sec 9, Badger Gap 7.5 Quad, Yakima Firing Center), Jeff Flemken, Linc Analysis, POB 684, Pullman, WA 99163	
0.999	1.000	0.999	0.978	0.946	0.991	0.982	0.996	0.986	0.986	217			WA	Mazama/6850	Sample #2 Toppenish Creek, T 9N R13E, SE 1/4, NW 1/4 of S2, G. Cleveland, Yakima Indian Nation	
1.000	0.977	0.994	0.979	0.964	0.995	0.962	1.000	0.985	0.985	216			WA	Mazama/6850	Sample #1, IHS Clinic near Yakima Indian Agency, G. Cleveland, Yakima Indian Nation	
0.998	0.977	0.994	0.979	0.982	1.000	0.941	1.000	0.985	0.985	856		29-Dec-94	OR	Mazama Climatic 6850 BP	Sample CH-7-5 from 150-180 cm depth, beach impounded lagoon N of Ana Springs in Summit Lake Basin, NW1/4, Sec. 36, T28S, R18W, Egl Rim 7.5 Quad, Dolly Fiedel, Dept. of Geosciences, Univ. of Missouri, Kansas City, MO	
1.000	0.953	0.999	0.978	0.964	0.986	0.974	0.996	0.985	0.985	457			OR	Mazama 6850 ?	Sample 35JE283, 398-2; Intotec, Willowdale Quad.	
0.996	0.977	0.994	0.978	0.952	0.991	0.981	0.996	0.985	0.985	192			ID	Mazama/6850	10BR790-545-10E-A, Pend Oreille River Site, Sandpoint, ID, C. Miss, NW Arch. Assoc. Inc.	
1.000	1.000	0.990	0.957	0.988	0.986	0.951	0.996	0.985	0.985	1108		3-Jan-97	WA	Mazama Climatic 6850 BP ??	Sample 45KT338 Tephra 2 (glass 1, 11 shards), depth 4.3m along Squaw Creek (SW1/4, NW1/4 of Sec 9, Badger Gap 7.5 Quad, Yakima Firing Center), Jeff Flemken, Linc Analysis, POB 684, Pullman, WA 99163	

number of records searched: 1145

WSU GeoAnalytical Laboratory

Analyses by electron microprobe

TABLE 1. GLASS CHEMISTRY OF FLATHEAD LAKE TEPHRAS

Oxide	FL-00-9P-III 45-46	FL-00-9P-III 78-79 bulk composition	glass1	glass 2	FL-00-9P-V 74-75
SiO ₂	73.09(0.34)	72.43(1.60)	69.08(0.60)	73.12(0.40)	77.31(0.15)
Al ₂ O ₃	14.50(0.23)	14.72(0.55)	15.79(0.06)	14.50(0.28)	12.80(0.14)
Fe ₂ O ₃	2.14(0.10)	2.34(0.48)	3.35(0.26)	2.13(0.06)	1.09(0.05)
TiO ₂	0.43(0.03)	0.46(0.08)	0.62(0.08)	0.43(0.02)	0.21(0.03)
Na ₂ O	4.81(0.16)	4.86(0.18)	4.95(0.22)	4.84(0.18)	3.52(0.10)
K ₂ O	2.68(0.10)	2.61(0.18)	2.25(0.02)	2.68(0.08)	3.42(0.08)
MgO	0.46(0.05)	0.54(0.18)	0.91(0.09)	0.46(0.03)	0.24(0.03)
CaO	1.67(0.15)	1.83(0.50)	2.87(0.28)	1.62(0.12)	1.22(0.04)
Cl	0.19(0.04)	0.18(0.03)	0.15(0.04)	0.19(0.02)	0.16(0.02)
Total**	100	100	100	100	100
Number of shards analyzed	14	18	3	15	16
Probable Source/Age	Mazama Climactic 6850 BP	Mazama Climactic 6850 BP	Mount St Helens T ? 6850 BP	Mazama Climactic 6850 BP	Glacier Peak 11,200 BP
Similarity Coefficient***	0.99	0.97	0.93	0.99	0.96+

* Standard deviations of the analyses given in parentheses

** Analyses normalized to 100 weight percent

*** Borchardt et al. (1972) J. Sed. Petrol., 42, 301-306

Tephra Glass Comparison

Sample name: Hendrix FL-00-9P-III, 7B-79 (glass 1)												
SiO ₂	TiO ₂	Al ₂ O ₃	MgO	CaO	BaO	MnO	Fe ₂ O ₃	Na ₂ O	K ₂ O	Cl	Total	
69.08	0.62	15.79	0.81	2.87	0.00	0.03	3.35	4.95	2.25	0.15	100.00	
1.00	0.25	1.00	0.25	1.00	0.00	0.00	1.00	1.00	1.00	weighting factor (only for oxides in bold type)		
Similarity Coefficients for 15 closest matches												
SiO ₂	TiO ₂	Al ₂ O ₃	MgO	CaO	Fe ₂ O ₃	Na ₂ O	K ₂ O	Cl	Total	weighted avg	sim coef	
0.996	0.935	0.994	0.934	0.892	0.988	0.948	0.978		0.964	1083	25-Sep-96	ID BP ??
0.992	0.954	0.976	0.912	0.906	0.970	0.967	0.966		0.960	908	10-Mar-95	MT 7000 BP
0.991	0.952	0.977	0.938	0.979	0.774	0.986	0.930		0.940	341	OR	7000 BP
0.989	0.969	0.959	0.901	0.843	0.980	0.998	0.852		0.937	1104	3-Jan-97	WA BP ??
0.986	0.861	0.998	0.850	1.000	0.755	0.972	0.924		0.833	212	WA	7000 BP
0.997	0.984	0.955	0.923	0.937	0.875	0.968	0.840		0.930	519	OR	
0.983	0.742	0.988	0.868	0.993	0.936	0.897	0.827		0.927	275	MT	SH Te ??
0.993	0.899	0.975	0.363	0.916	0.965	0.990	0.865		0.926	61	OR	MZ Liao Rock?7000
0.981	0.984	0.925	0.868	0.868	0.988	0.958	0.821		0.924	37	OR	7000 BP
0.980	0.677	0.995	0.791	0.983	0.886	0.905	0.900		0.922	374	WA	MSH T, 180
0.981	0.968	0.937	0.890	0.927	0.931	0.889	0.804		0.922	71	OR	
0.984	1.000	0.950	0.802	0.843	0.931	0.990	0.833		0.920	60	OR	MZ Liao Rock?7000
0.972	0.684	0.973	0.736	0.976	0.955	0.909	0.827		0.918	659	3-Sep-93	ID
0.984	0.849	0.959	0.934	0.801	0.946	0.957	0.852		0.916	912	10-Mar-95	MT 7000 BP
0.981	0.969	0.939	0.813	0.836	0.943	0.964	0.787		0.907	691	3-Nov-93	OR 120-130K BP
number of records searched:											1145	

Notes

Sample #95 (glass 3, 2 shards) from Bank Face #1, Slatum X, 81-82 cm bs, Site 10-BR-7000 572 along Pend Oreille River (NW1/4 Sec. 27, T56N, R5W), Christian Miss. NW Archaeological Associates, 54161/2 20th Avenue NW, Seattle, WA 98107

Sample S-2, depth =55cm, Unit 35/1E, Site 24LN1478, Yaak River, NE 1/4, Sec. 32, Bonnet Top 7.5 Quad, Montana, Matthew Zweifel, District Archaeologist, Three Rivers Ranger Dist, Kootenai NF, 1437 N. Hwy. 2, Troy, MT 59835

35WS120, TEU-2-444-1, Glass E, INFOTEC, North Central OR, SW of Sheniko

Sample 45KT338 Tephra 1 (glass 1, 3 shards), depth 4.3m along Squaw Creek (SW1/4, SW1/4 of Sec 9, Badger Gap 7.5 Quad, Yaldima Firing Center), Jeff Fienken, Lithic Analysts, POB 884, Pullman, WA 99163

POR-7-29-87, Tephra B, location unknown, E. Kiver, EWU

WL-A-9-1, Summer Lake Area, Wetland Levee Core Locality, P. Wigand, Desert Research Inst.

45PO137-5, Calispell Valley, MT, A. Thoms, Center for NW Anthropology

DR-97, Summer Lake Tephra Bed 6; Summer Lake, OR, Davis (1985) Quat. Res. 23 38-53

1911/AUG6, Malheur Wildlife Refuge, OR, J. Reese, Cultural Heritage Foundation

Sarna-Wojcicki et al., (1980) p 667-681 in Lipman and Mullineau eds. "The 1980 Eruptions of MSH, WA-USGS PP 1250

DR-18, Summer Lake Tephra Bed O, Summer Lake, OR; Davis (1985) Quat. Res. 23 38-53

GS-56, Summer Lake Tephra Bed 6; Summer Lake, OR, Davis (1985) Quat. Res. 23 38-53

Site #202, Sample#68, Glass 1, 68" depth in piston core in Bailey Bog, north end of Priest Lake, Idaho Alan Busacca, Dept. of Crop & Soil Science, Wash. State Univ.

Sample S-23, depth =68cm, Unit 19W/19S, Site 24LN1478, Yaak River, NE 1/4, Sec. 32, Bonnet Top 7.5 Quad, Montana, Matthew Zweifel, District Archaeologist, Three Rivers Ranger Dist, Kootenai NF, 1437 N. Hwy. 2, Troy, MT 59835

Sample 35H4914-TA-39, provided by InterMountain Research staff from Lake Malheur Area, OR, originally probed by J. Westgate and searched out by Andre as a Tulelake sample (SC-0.98), analyzed by us with even better results

Tephra Glass Comparison

Sample name: Hendrix FL-00-3P-III, 76-79 (bulk composition)														
SiO2	TiO2	Al2O3	MgO	CaO	BaO	MnO	Fe2O3	Na2O	K2O	Cl	Total			
72.43	0.46	14.72	0.54	1.83	0.00	0.03	2.34	4.86	2.61	0.18	100.00			
1.00	0.25	1.00	0.25	1.00	0.00	0.00	1.00	1.00	1.00	1.00	weighting factor (only for oxides in bold type)			
Similarity Coefficients for 15 closest matches														
SiO2	TiO2	Al2O3	MgO	CaO	Fe2O3	Na2O	K2O	Cl	weighted avg	sim coef	Source/Age	Date	State	Notes
0.999	0.958	0.978	1.000	0.934	0.987	0.946	0.970	0.970	0.970	0.970	Mazama 6850	421 S	OR	Westgate, Smith and Tomlinson (1970) in Early Man & Environments in NW North America, p.13-34, R.A. Smith & J.W. Smith eds.
0.993	0.913	0.983	0.870	0.891	0.996	0.998	0.986	0.970	0.970	0.970	MZ Climatic/6850 ?	88	OR	35KL972-795-13-3, Klamath Basin, 20 mi E of Klamath Falls, Jo Reese of Archaeological Invest. NW
0.995	0.913	0.982	0.870	0.918	0.983	0.976	0.989	0.968	0.968	0.968	Mazama, 6850 BP?	630	19-Jun-93 NV	Sample 2508 D-1, 8.2.1, Site 261, 2506 (depth = 54 cm) in Shoshone Range of North Central NV, (T32N, R47E, S24), Dan Dugas, Intermountain Research, Silver City, NV
0.994	0.935	0.981	0.889	0.951	0.979	0.981	0.946	0.967	0.967	0.967	Mazama Climatic 6850 BP ?	1093	26-Sep-96 WA	Sample Burbank Creek (glass 1, 11 shards), Yakima Canyon, between Yakima & Ellensburg, (NE 1/4, SE 1/4, Sec 4, T15N, R18E) Washington; Jodie Lamb, Department of Geology, Central Washington University, Ellensburg, WA
0.990	0.913	0.987	0.926	0.995	0.951	0.951	0.946	0.966	0.966	0.966	Tsuyawata (Crater Lake)-7000 BP	894	11-Jan-95 NV	Sample GS-34, Squaw Creek Terrace T1, 20km NW of Gerlach, NV; Davis (1983) Quaternary Research, 21, 312-324.
0.994	0.891	0.981	0.870	0.923	0.987	0.992	0.956	0.965	0.965	0.965	Mazama Climatic, 6850 BP ?	686	18-Oct-93 WA	Sample SO/61F-10-1 (upper ash), glass 1, depth 4.0m, Squaw Creek drainage, Yakima Training Center, K. Boreson, Archaeological & Historical Services, EWU
0.997	0.913	0.983	0.944	0.918	0.936	0.978	0.992	0.964	0.964	0.964	MZ climatic/6850 ?	98	OR	35KL972-795-24-1, Klamath Basin, 20 mi E of Klamath Falls, Jo Reese of Archaeological Invest. NW
0.994	0.935	0.985	0.982	0.902	0.953	0.977	0.963	0.964	0.964	0.964	Mazama Climatic 6850 BP	977	23-Aug-95 OR	Sample CH-7-5, lapilli from 2 m deep backhoe trench, 1 mile NW of Ana Springs Reservoir (shoreline in Summer Lake Basin (Lake Chewaucan), Dolly Freidel, Dept of Geography, Sonoma State Univ., Rohnert Park, CA 94928)
0.990	0.848	0.972	0.907	0.945	0.949	0.968	0.970	0.962	0.962	0.962	Mazama/6850	349	OR	Willowdale
0.995	0.978	0.983	0.889	0.869	0.996	0.972	0.970	0.962	0.962	0.962	Mazama/6850	306	ID	10BY309, TEU-2-115-2, INFOTEC, North Central Idaho near Meadow Creek
0.995	0.913	0.980	0.907	0.940	0.915	0.990	0.978	0.962	0.962	0.962	Mazama/6850	346	OR	Willowdale
0.994	0.935	0.986	0.981	0.885	0.953	0.984	0.970	0.962	0.962	0.962	Mazama Climatic 6850 BP	893	10-Jan-95 WA	Sample 621-8.1, depth = 3.2m bs, Foster Creek Drainage Basin, SE, SE, SE, Sec. 10, T16N, R21E, Boylston Quad., Stan Gough, Archaeological & Historical Services, EWU
0.991	0.870	0.979	0.833	0.896	0.970	1.000	0.985	0.961	0.961	0.961	Mazama/6850	234	WA	#2B (115cm), Beech Creek Archaeological Site, Packwood, WA, R. McClure, Gifford Pinchot NF
0.993	0.913	0.982	0.852	0.891	0.983	0.994	0.963	0.961	0.961	0.961	Mazama Climatic, 6850 BP ?	882	18-Oct-93 WA	Sample SE/045YA305-2, depth 4.0m, Selah Creek drainage, Yakima Training Center, K. Boreson, Archaeological & Historical Services, EWU
0.996	0.978	0.978	0.833	0.896	0.967	0.955	1.000	0.961	0.961	0.961	Mazama/6850	300	OR	35JE296, TEU-2-411-1, INFOTEC, North Central OR north of Willowdale

Number of records searched: 1145

WSU GeoAnalytical Laboratory

Analyses by electron microprobe

Sample name: Hendrix FL-00-9P-III, 78-79 (glass 2)

	SiO ₂	TiO ₂	Al ₂ O ₃	MgO	CaO	BaO	MnO	Fe ₂ O ₃	Na ₂ O	K ₂ O	Cl	Total
	73.12	0.43	14.50	0.46	1.62	0.00	0.03	2.13	4.84	0.19	100.00	
	1.00	0.25	1.00	0.25	1.00	0.00	0.00	1.00	1.00	1.00	weighting factor (only for oxides in bold type)	

Similarity Coefficients for 15 closest matches

sim coef

SiO ₂	TiO ₂	Al ₂ O ₃	MgO	CaO	Fe ₂ O ₃	Na ₂ O	K ₂ O	weighting factor	avg	max	Std	Date	State	Source/Age	Notes	weighted &
0.999	1.000	0.999	0.978	0.975	0.995	0.998	0.996	0.992	0.992	0.992	217	7-Mar-97	WA	Mazama/6850	Sample #2 Toppenish Creek, T 9N R13E, SE 1/4, NW 1/4 of S2, G. Cleveland, Yakima Indian Nation	Yakima
1.000	0.977	0.994	0.979	0.994	1.000	0.998	1.000	0.992	0.992	0.992	216		WA	Mazama/6850	Sample #1 IHS Clinic near Yakima Indian Agency, G. Cleveland, Yakima Indian Nation	
0.997	0.977	0.994	0.978	0.988	0.981	0.992	1.000	0.991	0.991	0.991	1036	7-Mar-97	WA	Mazama/6850 BP ??	Sample from sediments within rock shelter on upper Wapachuck River above Leavenworth, WA (Winlock 7.5 Quad, NW 1/4, NE 1/4 of Sec 21, T25N, R17E); Leavenworth, WA 99004	
0.999	0.930	0.999	1.000	0.994	0.995	0.992	0.989	0.991	0.991	0.991	188		ID	Mazama/6850	Sample from sediments within rock shelter on upper Wapachuck River above Leavenworth, WA (Winlock 7.5 Quad, NW 1/4, NE 1/4 of Sec 21, T25N, R17E); Leavenworth, WA 99004	
0.999	0.977	0.999	0.979	0.970	0.991	0.990	1.000	0.990	0.990	0.990	914	14-Apr-95	OR	Mazama Liao Rock 7000 BP	10/11/639 Pitsburg Landing, B. Cochran, Pitsburg Arch. Project, USFS	
0.998	0.966	0.999	0.978	0.988	0.986	0.996	0.985	0.990	0.990	0.990	778	26-Aug-94	OR	Mazama Climactic 6850 BP	Cloud Cap pumice collected by Mehninger, Wigand and Bacon on Aug 22, 1982 at Cloud Cap locality	
1.000	0.953	0.999	0.978	0.994	0.982	0.990	0.996	0.990	0.990	0.990	457		OR	Mazama 6850 ?	USGS Mazama Climactic Standard W-97, from Wasco OR, Andre Sama	
0.998	0.977	0.995	0.957	1.000	1.000	0.998	0.957	0.989	0.989	0.989	980	23-Aug-95	?	BP	Sample 35JE283, 398-2; infotec, Willowdale Quad.	
0.999	1.000	0.997	1.000	0.981	0.995	0.968	0.985	0.989	0.989	0.989	1017	12-Oct-95	OR	Mazama Climactic 6850 BP ???	Sample BB-6-1, Alan Busacca, Dept of Crop and Soil Science, WSU, Pullman, WA	
0.999	0.956	0.990	0.979	0.994	0.995	0.970	0.993	0.988	0.988	0.988	1086	26-Sep-96	WA	Mazama Climactic/Lao Rock 6850-7000 BP ??	MCA-2, depth=7cm on creek flood plain, Meyers Canyon near Mitchell, OR; 120 degrees and 6 and 44 degrees and 38; Dan Levis, Bur of Reclamation, Denver Federal Center, Denver, CO	
0.997	0.977	0.989	0.978	0.981	0.977	0.996	0.996	0.989	0.989	0.989	215		WA	Mazama/6850	Sample Eaton Ranch, Yakima Canyon between Yakima & Ellensburg, (NE1/4, SE1/4, Sec 4, T15N, R19E), Washington; Jodie Lamb, Department of Geology, Central Washington University, Ellensburg, WA	
0.997	1.000	0.983	0.978	0.988	0.996	0.976	1.000	0.988	0.988	0.988	798	16-Jun-94	WA	Mazama Climactic 6850 BP ?	Sample from along Skagit River near Sky Creek Siding northern Skagit Co., J. Reidel, N Cascades NP	
0.998	1.000	0.997	1.000	0.984	0.964	0.976	0.993	0.988	0.988	0.988	319		OR	Mazama/6850	KL-23/424, eastern end of Miller Island in the Columbia River near Hell's Gate (mile 205), John Dougherty, Archaeological Services Inc., Stockton, CA	
0.999	0.953	0.995	0.978	0.988	0.986	0.984	0.985	0.988	0.988	0.988	19		OR	Mazama/6850	35JE51B, SOX-6-T30-1, Ash #1, INFOPEC, North Central OR north of Willowdale Twin Lakes 1.840m, Wallowa-Whitman NF, OR, Marden-Mehninger	
0.997	0.977	0.994	0.978	0.981	0.986	0.975	0.996	0.988	0.988	0.988	192		ID	Mazama/6850	108R790-545-10E-A, Pend Oreille River Site, Sandpoint, ID, C. Miss, NW Arch. Assoc. Inc.	

number of records searched: 1145



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Beta Analytic Inc.
4985 SW 74 Court
Miami, Florida 33155 USA
Tel: 305 667 5167
Fax: 305 663 0964
Beta@radiocarbon.com
www.radiocarbon.com

Dr. MURRY A. TAMERS
Mr. DARDEN G. HOOD
Directors

Mr. RONALD E. HATFIELD
Mr. CHRISTOPHER L. PATRICK
Deputy Directors

ANALYTICAL PROCEDURES AND FINAL REPORT

Final Report

The final report package includes the final date report, a statement outlining our analytical procedures, a glossary of pretreatment terms, calendar calibration information, billing documents (containing balance/credit information and the number of samples submitted within the yearly discount period), and peripheral items to use with future submittals. The final report includes the individual analysis method, the delivery basis, the material type and the individual pretreatments applied. The final report will be sent by mail, fax or e-mail, where available.

Pretreatment

Pretreatment methods are reported along with each result. All necessary chemical and mechanical pretreatments of the submitted material are applied at the laboratory to isolate ^{14}C which may best represent the time event of interest. When interpreting the results, it is important to consider the pretreatments. Some samples cannot be fully pretreated, making their ^{14}C ages more subjective than samples which can be fully pretreated. Some materials receive no pretreatments. Please read the pretreatment glossary.

Analysis

Materials measured by the radiometric technique are analyzed by synthesizing sample carbon to benzene (92% C), measuring for ^{14}C content in a scintillation spectrometer, and then calculating for radiocarbon age. If the Extended Counting Service is used, the ^{14}C content is measured for a greatly extended period of time. AMS results are derived from reduction of sample carbon to graphite (100 %C), along with standards and backgrounds. The graphite is then detected for ^{14}C content in an accelerator-mass-spectrometer (AMS) located at one of 9 collaborating research facilities, who return the raw data to us for verification, isotopic fractionation correction, calculation calendar calibration, and reporting.

The Radiocarbon Age and Calendar Calibration

The "Conventional ^{14}C Age (*)" is the result after applying $^{13}\text{C}/^{12}\text{C}$ corrections to the measured age and is the most appropriate radiocarbon age (the "*" is discussed at the bottom of the final report). Applicable calendar calibrations are included for materials 0 and about 20,000 BP. If certain calibrations are not included with a report, the results were either too young, too old, or inappropriate for calibration.

**BETA ANALYTIC INC.
RADIOCARBON DATING LABORATORY
CALIBRATED C-14 DATING RESULTS**

Calibrations of radiocarbon age determinations are applied to convert BP results to calendar years. The short term difference between the two is caused by fluctuations in the heliomagnetic modulation of the galactic cosmic radiation and, recently, large scale burning of fossil fuels and nuclear devices testing. Geomagnetic variations are the probable cause of longer term differences.

The parameters used for the corrections have been obtained through precise analyses of hundreds of samples taken from known-age tree rings of oak, sequoia, and fir up to about 10,000 BP. Calibration using tree-rings to about 12,000 BP is still being researched and provides somewhat less precise correlation. Beyond that, up to about 20,000 BP, correlation using a modeled curve determined from U/Th measurements on corals is used. This data is still highly subjective. Calibrations are provided up to about 19,000 years BP using the most recent calibration data available (Radiocarbon, Vol 40, No. 3, 1998).

The Pretoria Calibration Procedure (Radiocarbon, Vol 35, No. 1, 1993, pg 317) program has been chosen for these calendar calibrations. It uses splines through the tree-ring data as calibration curves, which eliminates a large part of the statistical scatter of the actual data points. The spline calibration allows adjustment of the average curve by a quantified closeness-of-fit parameter to the measured data points. A single spline is used for the precise correlation data available back to 9900 BP for terrestrial samples and about 6900 BP for marine samples. Beyond that, splines are taken on the error limits of the correlation curve to account for the lack of precision in the data points.

In describing our calibration curves, the solid bars represent one sigma statistics (68% probability) and the hollow bars represent two sigma statistics (95% probability). Marine carbonate samples that have been corrected for $\delta^{13}C/^{12}C$, have also been corrected for both global and local geographic reservoir effects (as published in Radiocarbon, Volume 35, Number 1, 1993) prior to the calibration. Marine carbonates that have not been corrected for $\delta^{13}C/^{12}C$ are adjusted by an assumed value of 0 ‰ in addition to the reservoir corrections. Reservoir corrections for fresh water carbonates are usually unknown and are generally not accounted for in those calibrations. In the absence of measured $\delta^{13}C/^{12}C$ ratios, a typical value of -5 ‰ is assumed for freshwater carbonates.

(Caveat: the correlation curve for organic materials assume that the material dated was living for exactly ten years (e.g. a collection of 10 individual tree rings taken from the outer portion of a tree that was cut down to produce the sample in the feature dated). For other materials, the maximum and minimum calibrated age ranges given by the computer program are uncertain. The possibility of an "old wood effect" must also be considered, as well as the potential inclusion of younger or older material in matrix samples. Since these factors are indeterminant error in most cases, these calendar calibration results should be used only for illustrative purposes. In the case of carbonates, reservoir correction is theoretical and the local variations are real, highly variable and dependant on provenience. Since imprecision in the correlation data beyond 10,00 years is high, calibrations in this range are likely to change in the future with refinement in the correlation curve. The age ranges and especially the intercept ages generated by the program, must be considered as approximations.)

PRETREATMENT GLOSSARY

Pretreatment of submitted materials is required to eliminate secondary carbon components. These components, if not eliminated, could result in a radiocarbon date which is too young or too old. Pretreatment does not ensure that the radiocarbon date will represent the time event of interest. This is determined by the sample integrity. The old wood effect, burned intrusive roots, bioturbation, secondary deposition, secondary biogenic activity incorporating recent carbon (bacteria) and the analysis of multiple components of differing age are just some examples of potential problems. The pretreatment philosophy is to reduce the sample to a single component, where possible, to minimize the added subjectivity associated with these types of problems.

"acid/alkali/acid"

The sample was first gently crushed/dispersed in deionized water. It was then given hot HCl acid washes to eliminate carbonates and alkali washes (NaOH) to remove secondary organic acids. The alkali washes were followed by a final acid rinse to neutralize the solution prior to drying. Chemical concentrations, temperatures, exposure times, and number of repetitions, were applied accordingly with the uniqueness of the sample. Each chemical solution was neutralized prior to application of the next. During these serial rinses, mechanical contaminants such as associated sediments and rootlets were eliminated. This type of pretreatment is considered a "full pretreatment". On occasion the report will list the pretreatment as "acid/alkali/acid - insolubles" to specify which fraction of the sample was analyzed. This is done on occasion with sediments (See "acid/alkali/acid - solubles")

Typically applied to: charcoal, wood, some peats, some sediments, textiles

"acid/alkali/acid - solubles"

On occasion the alkali soluble fraction will be analyzed. This is a special case where soil conditions imply that the soluble fraction will provide a more accurate date. It is also used on some occasions to verify the present/absence or degree of contamination present from secondary organic acids. The sample was first pretreated with acid to remove any carbonates and to weaken organic bonds. After the alkali washes (as discussed above) are used, the solution containing the alkali soluble fraction is isolated/filtered and combined with acid. The soluble fraction which precipitates is rinsed and dried prior to combustion.

"acid washes"

Surface area was increased as much as possible. Solid chunks were crushed, fibrous materials were shredded, and sediments were dispersed. Acid (HCl) was applied repeatedly to ensure the absence of carbonates. Chemical concentrations, temperatures, exposure times, and number of repetitions, were applied accordingly with the uniqueness of each sample. The sample, for a number of reasons, could not be subjected to alkali washes to ensure the absence of secondary organic acids. The most common reason is that the primary carbon is soluble in the alkali. Dating results reflect the total organic content of the analyzed material. Their accuracy depends on the researcher's ability to subjectively eliminate potential contaminants based on contextual facts.

Typically applied to: organic sediments, some peats, small wood or charcoal, special cases

"collagen extraction"

The material was first tested for friability ("softness"). Very soft bone material is an indication of the potential absence of the collagen fraction (basal bone protein acting as a "reinforcing agent" within the crystalline apatite structure). It was then washed in de-ionized water and gently crushed. Dilute, cold HCl acid was repeatedly applied and replenished until the mineral fraction (bone apatite) was eliminated. The collagen was then dissected and inspected for rootlets. Any rootlets present were also removed when replenishing the acid solutions. Where possible, usually dependant on the amount of collagen available, alkali (NaOH) was also applied to ensure the absence of secondary organic acids.

Typically applied to: bones

"acid etch"

The calcareous material was first washed in de-ionized water, removing associated organic sediments and debris (where present). The material was then crushed/dispersed and repeatedly subjected to HCl etches to eliminate secondary carbonate components. In the case of thick shells, the surfaces were physically abraded prior to etching down to a hard, primary core remained. In the case of porous carbonate nodules and caliche, very long exposure times were applied to allow infiltration of the acid. Acid exposure times, concentrations, and number of repetitions, were applied accordingly with the uniqueness of the sample.

Typically applied to: shells, caliche, calcareous nodules

"neutralized"

Carbonates precipitated from ground water are usually submitted in an alkaline condition (ammonium hydroxide or sodium hydroxide solution). Typically this solution is neutralized in the original sample container, using deionized water. If larger volume dilution was required, the precipitate and solution were transferred to a sealed separatory flask and rinsed to neutrality. Exposure to atmosphere was minimal.

Typically applied to: Strontium carbonate, Barium carbonate
(i.e. precipitated ground water samples)

"none"

No laboratory pretreatments were applied. Special requests and pre-laboratory pretreatment usually accounts for this.

"acid/alkali/acid/cellulose extraction"

Following full acid/alkali/acid pretreatments, the sample is rinsed in NaClO₂ under very controlled conditions (Ph = 3, temperature = 70 degrees C). This eliminates all components except wood cellulose. It is useful for woods which are either very old or highly contaminated.

Applied to: wood

"carbonate precipitation"

Dissolved carbon dioxide and carbonate species are precipitated from submitted water by complexing them as ammonium carbonate. Strontium chloride is added to the ammonium carbonate solution and strontium carbonate is precipitated for the analysis. The result is representative of the dissolved inorganic carbon within the water. Results are reported as "water DIC".

Applied to: water

**BETA ANALYTIC INC.**

DR. M.A. TAMERS and MR. D.G. HOOD

UNIVERSITY BRANCH
4985 S.W. 74 COURT
MIAMI, FLORIDA, USA 33155
PH: 305/667-5167 FAX: 305/663-0964
E-MAIL: beta@radiocarbon.com

REPORT OF RADIOCARBON DATING ANALYSES

Mr. Michael Sperazza

Report Date: 3/28/2003

University of Montana

Material Received: 2/24/2003

Sample Data	Measured Radiocarbon Age	$^{13}\text{C}/^{12}\text{C}$ Ratio	Conventional Radiocarbon Age(*)
Beta - 176803 SAMPLE : FL009PV74 ANALYSIS : AMS-Standard delivery MATERIAL/PRETREATMENT : (organic sediment): acid washes 2 SIGMA CALIBRATION : Cal BC 14090 to 13340 (Cal BP 16040 to 15280)	13090 +/- 40 BP	-26.4 o/oo	13070 +/- 40 BP

Dates are reported as RCYBP (radiocarbon years before present, "present" = 1950A.D.). By international convention, the modern reference standard was 95% of the C^{14} content of the National Bureau of Standards' Oxalic Acid & calculated using the Libby C^{14} half life (5568 years). Quoted errors represent 1 standard deviation statistics (68% probability) & are based on combined measurements of the sample, background, and modern reference standards.

Measured $\text{C}^{13}/\text{C}^{12}$ ratios were calculated relative to the PDB-1 international standard and the RCYBP ages were normalized to -25 per mil. If the ratio and age are accompanied by an (*), then the $\text{C}^{13}/\text{C}^{12}$ value was estimated, based on values typical of the material type. The quoted results are NOT calibrated to calendar years. Calibration to calendar years should be calculated using the Conventional C^{14} age.

CALIBRATION OF RADIOCARBON AGE TO CALENDAR YEARS

(Variables: C13/C12=-26.4;lab. mult=1)

Laboratory number: Beta-176803

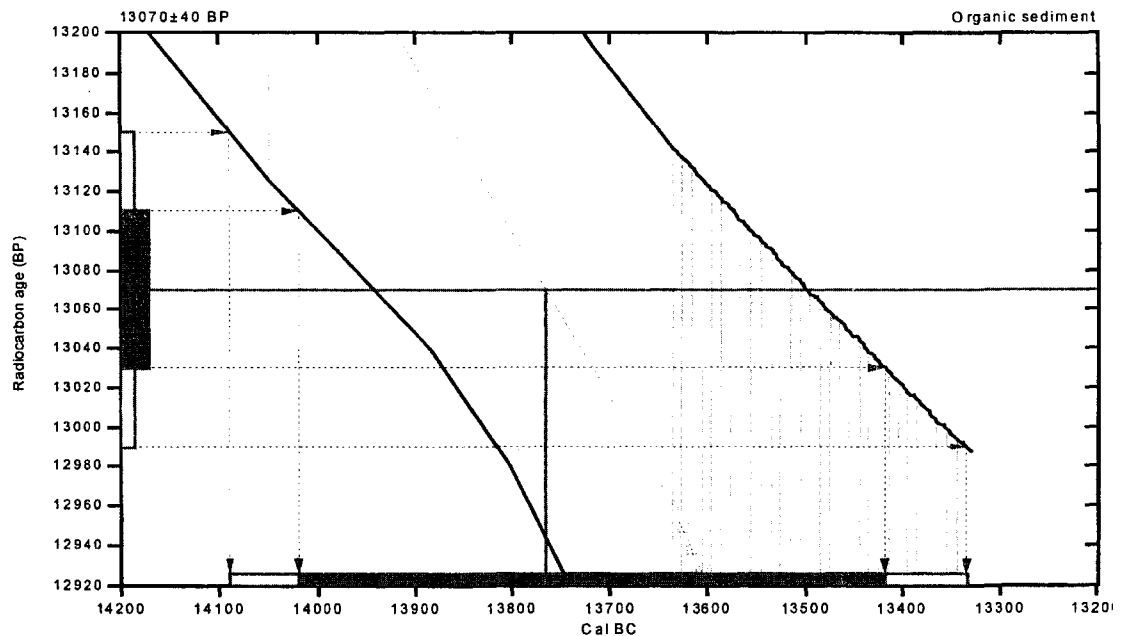
Conventional radiocarbon age: 13070±40 BP

2 Sigma calibrated result: Cal BC 14090 to 13340 (Cal BP 16040 to 15280)
(95% probability)

Intercept data

Intercept of radiocarbon age
with calibration curve: Cal BC 13760 (Cal BP 15720)

1 Sigma calibrated result: Cal BC 14020 to 13420 (Cal BP 15970 to 15370)
(68% probability)



References:

Database used

Calibration Database

Editorial Comment

Stuiver, M., van der Plicht, H., 1998, Radiocarbon 40(3), pxi-xiii
INTCAL98 Radiocarbon Age Calibration

Stuiver, M., et. al., 1998, Radiocarbon 40(3), p1041-1083

Mathematics

A Simplified Approach to Calibrating C14 Dates

Talma, A. S., Vogel, J. C., 1993, Radiocarbon 35(2), p317-322

Spl. fit

*includes
Tree ring error*

*Tends
to be more
conservative
10yr
fandly.*

Beta Analytic Inc.

4985 SW 74 Court, Miami, Florida 33155 USA • Tel: (305) 667 5167 • Fax: (305) 663 0964 • E-Mail: beta@radiocarbon.com

**BETA ANALYTIC INC.**

DR. M.A. TAMERS and MR. D.G. HOOD

UNIVERSITY BRANCH
4985 S.W. 74 COURT
MIAMI, FLORIDA, USA 33155
PH: 305/667-5167 FAX: 305/663-0964
E-MAIL: beta@radiocarbon.com

REPORT OF RADIOCARBON DATING ANALYSES

Mr. Michael Sperazza

Report Date: 10/21/2003

University of Montana

Material Received: 9/25/2003

Sample Data	Measured Radiocarbon Age	¹³ C/ ¹² C Ratio	Conventional Radiocarbon Age(*)
Beta - 183410 SAMPLE : FL0315K-195 ANALYSIS : AMS-Standard delivery MATERIAL/PRETREATMENT : (wood): acid/alkali/acid 2 SIGMA CALIBRATION : Cal BC 5840 to 5710 (Cal BP 7790 to 7660)	6950 +/- 40 BP	-28.7 o/oo	6890 +/- 40 BP
Beta - 183411 SAMPLE : FL0315K-II69 ANALYSIS : AMS-Standard delivery MATERIAL/PRETREATMENT : (wood): acid/alkali/acid 2 SIGMA CALIBRATION : Cal BC 8150 to 8140 (Cal BP 10100 to 10090) AND Cal BC 7970 to 7720 (Cal BP 9920 to 9660)	8820 +/- 40 BP	-27.4 o/oo	8780 +/- 40 BP
Beta - 183412 SAMPLE : FL0316K-144 ANALYSIS : AMS-Standard delivery MATERIAL/PRETREATMENT : (wood): acid/alkali/acid 2 SIGMA CALIBRATION : Cal BC 3340 to 2930 (Cal BP 5290 to 4880)	4430 +/- 40 BP	-23.8 o/oo	4450 +/- 40 BP
Beta - 183413 SAMPLE : FL0316KIII63 ANALYSIS : AMS-Standard delivery MATERIAL/PRETREATMENT : (wood): acid/alkali/acid 2 SIGMA CALIBRATION : Cal BC 7030 to 6860 (Cal BP 8980 to 8820) AND Cal BC 6850 to 6650 (Cal BP 8800 to 8600)	7920 +/- 40 BP	-26.1 o/oo	7900 +/- 40 BP
Beta - 183414 SAMPLE : FL0316K-IV27 ANALYSIS : AMS-Standard delivery MATERIAL/PRETREATMENT : (wood): acid/alkali/acid 2 SIGMA CALIBRATION : Cal BC 8290 to 8210 (Cal BP 10240 to 10160)	9030 +/- 50 BP	-24.3 o/oo	9040 +/- 50 BP

Dates are reported as RCYBP (radiocarbon years before present, "present" = 1950 A.D.). By international convention, the modern reference standard was 95% of the C14 content of the National Bureau of Standards' Oxalic Acid & calculated using the Libby C14 half life (5568 years). Quoted errors represent 1 standard deviation statistics (68% probability) & are based on combined measurements of the sample, background, and modern reference standards.

Measured C13/C12 ratios were calculated relative to the PDB-1 international standard and the RCYBP ages were normalized to -25 per mil. If the ratio and age are accompanied by an (*), then the C13/C12 value was estimated, based on values typical of the material type. The quoted results are NOT calibrated to calendar years. Calibration to calendar years should be calculated using the Conventional C14 age.

**BETA ANALYTIC INC.**

DR. M.A. TAMERS and MR. D.G. HOOD

UNIVERSITY BRANCH

4985 S.W. 74 COURT

MIAMI, FLORIDA, USA 33155

PH: 305/667-5167 FAX: 305/663-0964

E-MAIL: beta@radiocarbon.com

REPORT OF RADIOCARBON DATING ANALYSES

Mr. Michael Sperazza

Report Date: 10/21/2003

Sample Data	Measured Radiocarbon Age	$^{13}\text{C}/^{12}\text{C}$ Ratio	Conventional Radiocarbon Age(*)
Beta - 183415 SAMPLE : FL0316K-V20 ANALYSIS : AMS-Standard delivery MATERIAL/PRETREATMENT : (wood): acid/alkali/acid 2 SIGMA CALIBRATION : Cal BC 10820 to 10800 (Cal BP 12760 to 12740) AND Cal BC 10700 to 10510 (Cal BP 12650 to 12460) Cal BC 10450 to 9950 (Cal BP 12400 to 11900)	10320 +/- 50 BP	-23.7 o/oo	10340 +/- 50 BP
Beta - 183416 SAMPLE : FL0319K-IV44 ANALYSIS : AMS-Standard delivery MATERIAL/PRETREATMENT : (wood): acid/alkali/acid 2 SIGMA CALIBRATION : Cal BC 13360 to 12700 (Cal BP 15310 to 14650) AND Cal BC 12420 to 12130 (Cal BP 14370 to 14080) Cal BC 11950 to 11920 (Cal BP 13900 to 13870)	12220 +/- 50 BP	-24.6 o/oo	12230 +/- 50 BP

Dates are reported as RCYBP (radiocarbon years before present, "present" = 1950 A.D.). By international convention, the modern reference standard was 95% of the C14 content of the National Bureau of Standards' Oxalic Acid & calculated using the Libby C14 half life (5568 years). Quoted errors represent 1 standard deviation statistics (68% probability) & are based on combined measurements of the sample, background, and modern reference standards.

Measured C13/C12 ratios were calculated relative to the PDB-1 international standard and the RCYBP ages were normalized to -25 per mil. If the ratio and age are accompanied by an (*), then the C13/C12 value was estimated, based on values typical of the material type. The quoted results are NOT calibrated to calendar years. Calibration to calendar years should be calculated using the Conventional C14 age.

CALIBRATION OF RADIOCARBON AGE TO CALENDAR YEARS

(Variables: C13/C12=-28.7;lab. mult=1)

Laboratory number: **Beta-183410**

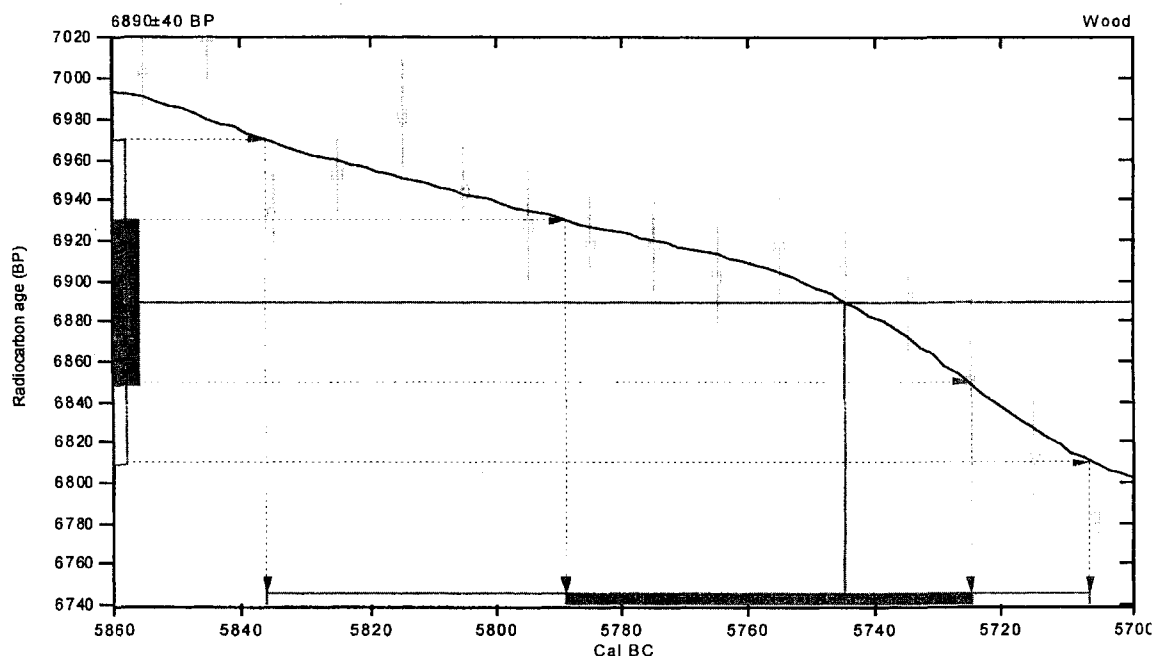
Conventional radiocarbon age: **6890±40 BP**

2 Sigma calibrated result: Cal BC 5840 to 5710 (Cal BP 7790 to 7660)
(95% probability)

Intercept data

Intercept of radiocarbon age
with calibration curve: Cal BC 5740 (Cal BP 7700)

1 Sigma calibrated result: Cal BC 5790 to 5720 (Cal BP 7740 to 7680)
(68% probability)



References:

Database used

INTCAL98

Calibration Database

Editorial Comment

Stuiver, M., van der Plicht, H., 1998, *Radiocarbon* 40(3), pxii-xiii

INTCAL98 Radiocarbon Age Calibration

Stuiver, M., et al., 1998, *Radiocarbon* 40(3), p1041-1083

Mathematics

A Simplified Approach to Calibrating C14 Dates

Talma, A. S., Vogel, J. C., 1993, *Radiocarbon* 35(2), p317-322

Beta Analytic Radiocarbon Dating Laboratory

4985 S.W. 74th Court, Miami, Florida 33155 • Tel: (305) 667-5167 • Fax: (305) 663-0964 • E-Mail: beta@radiocarbon.com

CALIBRATION OF RADIOCARBON AGE TO CALENDAR YEARS

(Variables: C13/C12=-23.8;lab. mult=1)

Laboratory number: **Beta-183412**

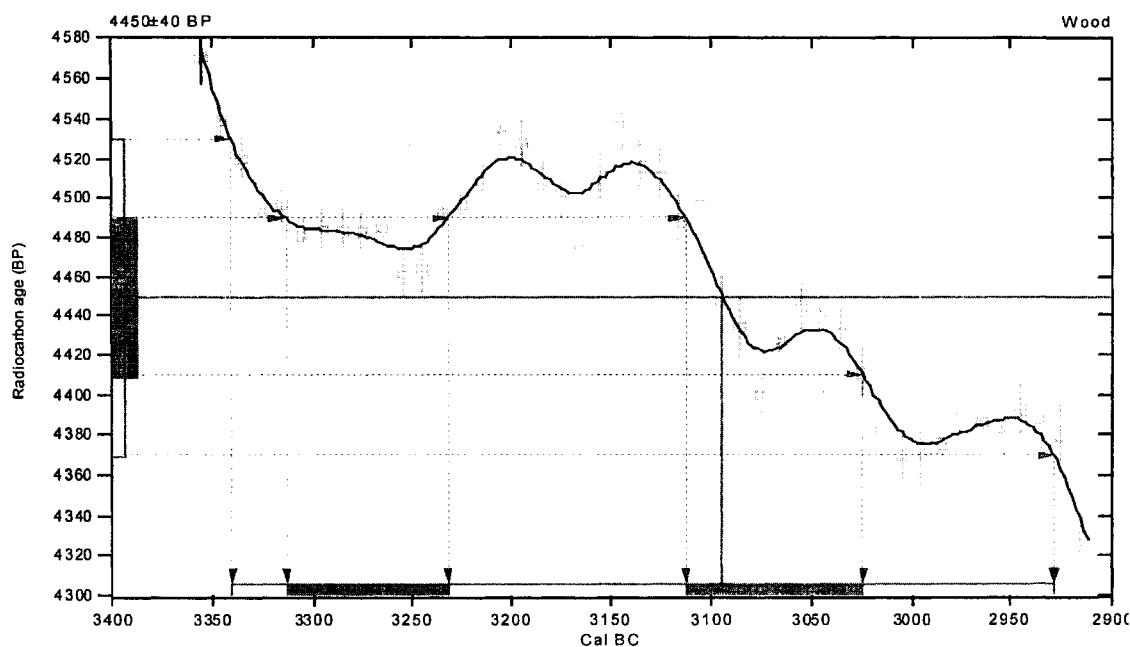
Conventional radiocarbon age: **4450±40 BP**

2 Sigma calibrated result: Cal BC 3340 to 2930 (Cal BP 5290 to 4880)
(95% probability)

Intercept data

Intercept of radiocarbon age
with calibration curve: Cal BC 3090 (Cal BP 5040)

1 Sigma calibrated results: Cal BC 3310 to 3230 (Cal BP 5260 to 5180) and
(68% probability) **Cal BC 3110 to 3020 (Cal BP 5060 to 4970)**



References:

Database used

INTCAL98

Calibration Database

Editorial Comment

Stuiver, M., van der Plicht, H., 1998, Radiocarbon 40(3), pxi-xiii

INTCAL98 Radiocarbon Age Calibration

Stuiver, M., et. al., 1998, Radiocarbon 40(3), p1041-1083

Mathematics

A Simplified Approach to Calibrating C14 Dates

Talma, A. S., Vogel, J. C., 1993, Radiocarbon 35(2), p317-322

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4985 S.W. 74th Court, Miami, Florida 33155 • Tel: (305) 667-5167 • Fax: (305) 663-0964 • E-Mail: beta@radiocarbon.com

CALIBRATION OF RADIOCARBON AGE TO CALENDAR YEARS

(Variables: C13/C12=-26.1;lab. mult=1)

Laboratory number: **Beta-183413**

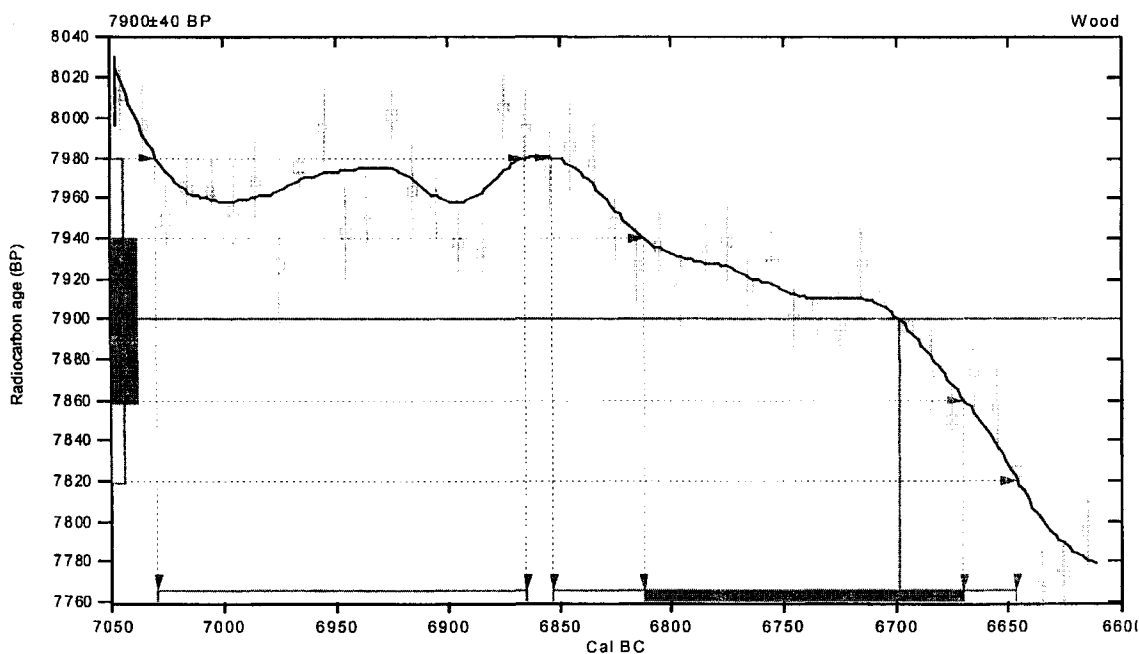
Conventional radiocarbon age: **7900±40 BP**

2 Sigma calibrated results: **Cal BC 7030 to 6860 (Cal BP 8980 to 8820) and**
(95% probability) Cal BC 6850 to 6650 (Cal BP 8800 to 8600)

Intercept data

Intercept of radiocarbon age
 with calibration curve: **Cal BC 6700 (Cal BP 8650)**

1 Sigma calibrated result: **Cal BC 6810 to 6670 (Cal BP 8760 to 8620)**
(68% probability)



References:

Database used

INTCAL98

Calibration Database

Editorial Comment

Stuiver, M., van der Plicht, H., 1998, Radiocarbon 40(3), pxii-xiii

INTCAL98 Radiocarbon Age Calibration

Stuiver, M., et. al., 1998, Radiocarbon 40(3), p1041-1083

Mathematics

A Simplified Approach to Calibrating C14 Dates

Talma, A. S., Vogel, J. C., 1993, Radiocarbon 35(2), p317-322

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4985 S.W. 74th Court, Miami, Florida 33155 • Tel: (305) 667-5167 • Fax: (305) 663-0964 • E-Mail: beta@radiocarbon.com

CALIBRATION OF RADIOCARBON AGE TO CALENDAR YEARS

(Variables: C13/C12=-24.3:lab. mult=1)

Laboratory number: Beta-183414

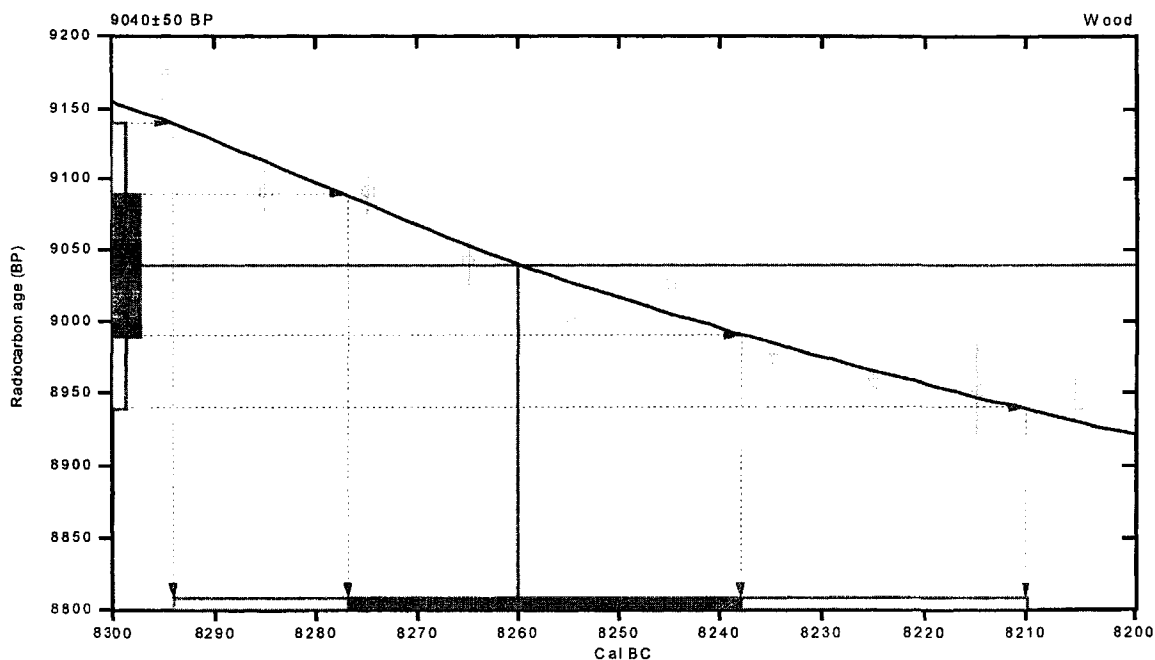
Conventional radiocarbon age: 9040±50 BP

2 Sigma calibrated result: Cal BC 8290 to 8210 (Cal BP 10240 to 10160)
(95% probability)

Intercept data

Intercept of radiocarbon age
with calibration curve: Cal BC 8260 (Cal BP 10210)

1 Sigma calibrated result: Cal BC 8280 to 8240 (Cal BP 10230 to 10190)
(68% probability)



References:

Database used

Calibration Database

Editorial Comment

Stuiver, M., van der Plicht, H., 1998, *Radiocarbon* 40(3), pxii-xiii

INTCAL98 Radiocarbon Age Calibration

Stuiver, M., et. al., 1998, *Radiocarbon* 40(3), p1041-1083

Mathematics

A Simplified Approach to Calibrating C14 Dates

Talma, A. S., Vogel, J. C., 1993, *Radiocarbon* 35(2), p317-322

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CALIBRATION OF RADIOCARBON AGE TO CALENDAR YEARS

(Variables: C13/C12=-23.7;lab. mult=1)

Laboratory number: Beta-183415

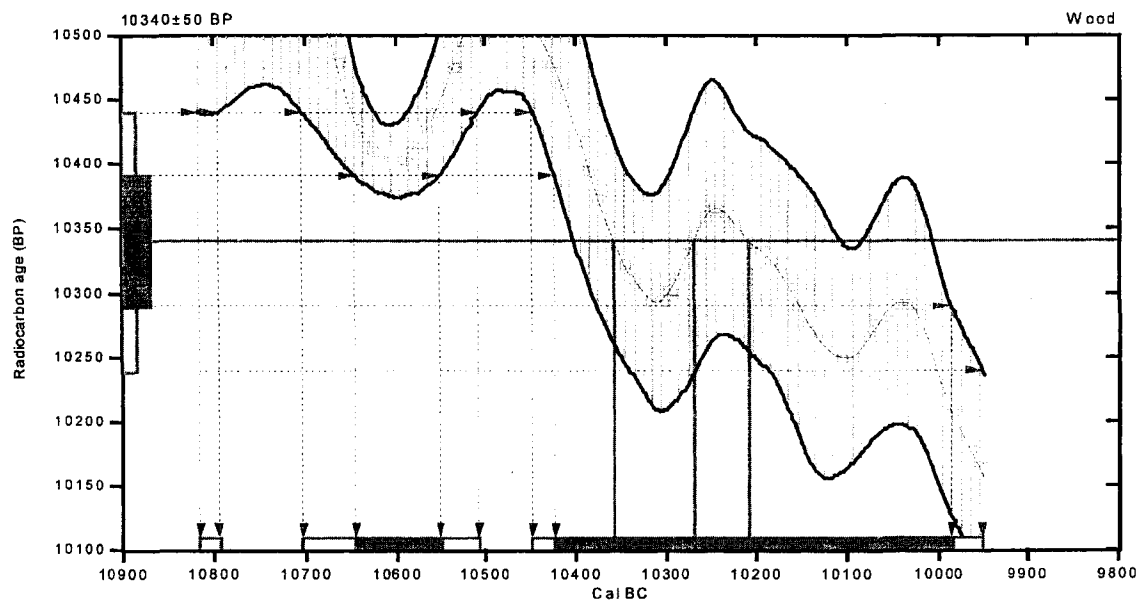
Conventional radiocarbon age: 10340±50 BP

2 Sigma calibrated results: Cal BC 10820 to 10800 (Cal BP 12760 to 12740) and
(95% probability) Cal BC 10700 to 10510 (Cal BP 12650 to 12460) and
Cal BC 10450 to 9950 (Cal BP 12400 to 11900)

Intercept data

Intercepts of radiocarbon age
with calibration curve: Cal BC 10360 (Cal BP 12310) and
Cal BC 10270 (Cal BP 12220) and
Cal BC 10210 (Cal BP 12160)

1 Sigma calibrated results: Cal BC 10640 to 10550 (Cal BP 12600 to 12500) and
(68% probability) Cal BC 10420 to 9990 (Cal BP 12370 to 11940)



References:

Database used

Calibration Database

Editorial Comment

Stuiver, M., van der Plicht, H., 1998, *Radiocarbon* 40(3), p. xii-xiii

INTCAL98 Radiocarbon Age Calibration

Stuiver, M., et. al., 1998, *Radiocarbon* 40(3), p. 1041-1083

Mathematics

A Simplified Approach to Calibrating C14 Dates

Talma, A. S., Vogel, J. C., 1993, *Radiocarbon* 35(2), p. 317-322

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4985 SW 74 Court, Miami, Florida 33155 USA • Tel: (305) 667 5167 • Fax: (305) 663 0964 • E-Mail: beta@radiocarbon.com

CALIBRATION OF RADIOCARBON AGE TO CALENDAR YEARS

(Variables: C13/C12=-24.6;lab. mult=1)

Laboratory number: Beta-183416

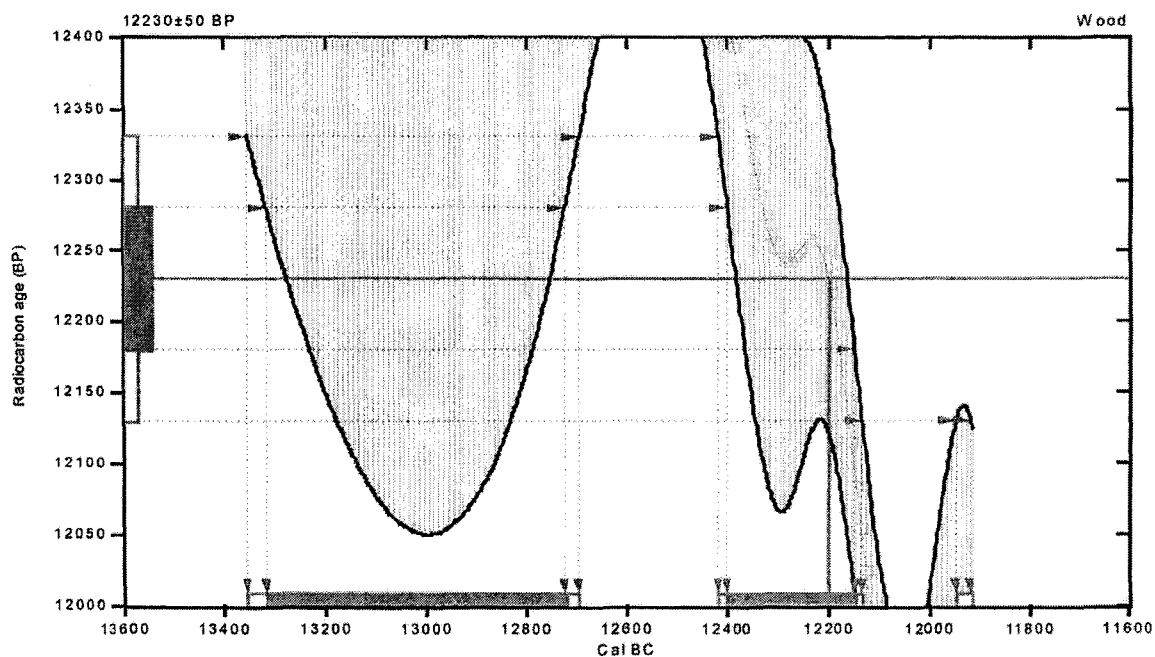
Conventional radiocarbon age: 12230±50 BP

2 Sigma calibrated results: Cal BC 13360 to 12700 (Cal BP 15310 to 14650) and
(95% probability) Cal BC 12420 to 12130 (Cal BP 14370 to 14080) and
Cal BC 11950 to 11920 (Cal BP 13900 to 13870)

Intercept data

Intercept of radiocarbon age
with calibration curve: Cal BC 12200 (Cal BP 14150)

1 Sigma calibrated results: Cal BC 13320 to 12720 (Cal BP 15270 to 14670) and
(68% probability) Cal BC 12400 to 12150 (Cal BP 14350 to 14100)



References:

Database used

Calibration Database

Editorial Comment

Stuiver, M., van der Plicht, H., 1998, *Radiocarbon* 40(3), pxi-xiii

INTCAL98 Radiocarbon Age Calibration

Stuiver, M., et. al., 1998, *Radiocarbon* 40(3), p1041-1083

Mathematics

A Simplified Approach to Calibrating C14 Dates

Talma, A. S., Vogel, J. C., 1993, *Radiocarbon* 35(2), p317-322

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**BETA ANALYTIC INC.**

DR. M.A. TAMERS and MR. D.G. HOOD

UNIVERSITY BRANCH
4985 S.W. 74 COURT
MIAMI, FLORIDA, USA 33155
PH: 305/667-5167 FAX: 305/663-0964
E-MAIL: beta@radiocarbon.com

REPORT OF RADIOCARBON DATING ANALYSES

Mr. Michael Sperazza

Report Date: 10/24/2003

University of Montana

Material Received: 10/15/2003

Sample Data	Measured Radiocarbon Age	$^{13}\text{C}/^{12}\text{C}$ Ratio	Conventional Radiocarbon Age(*)
Beta - 184123 SAMPLE: FL0315K-144 ANALYSIS: AMS-Standard delivery MATERIAL/PRETREATMENT: (wood): acid/alkali/acid 2 SIGMA CALIBRATION: Cal BC 1210 to 920 (Cal BP 3160 to 2870) Comment: the original sample was too small for a $^{13}\text{C}/^{12}\text{C}$ ratio measurement. However, a ratio including both natural and laboratory effects was measured during the ^{14}C detection to derive a Conventional Radiocarbon Age, suitable for applicable calendar calibration.	NA	NA	2880 +/- 50 BP

Dates are reported as RCYBP (radiocarbon years before present, "present" = 1950 A.D.). By international convention, the modern reference standard was 95% of the C^{14} content of the National Bureau of Standards' Oxalic Acid & calculated using the Libby C^{14} half life (5568 years). Quoted errors represent 1 standard deviation statistics (68% probability) & are based on combined measurements of the sample, background, and modern reference standards.

Measured $\text{C}^{13}/\text{C}^{12}$ ratios were calculated relative to the PDB-1 international standard and the RCYBP ages were normalized to -25 per mil. If the ratio and age are accompanied by an (*), then the $\text{C}^{13}/\text{C}^{12}$ value was estimated, based on values typical of the material type. The quoted results are NOT calibrated to calendar years. Calibration to calendar years should be calculated using the Conventional C^{14} age.

CALIBRATION OF RADIOCARBON AGE TO CALENDAR YEARS

(Variables: C13/C12=-25;lab. mult=1)

Laboratory number: **Beta-184123**

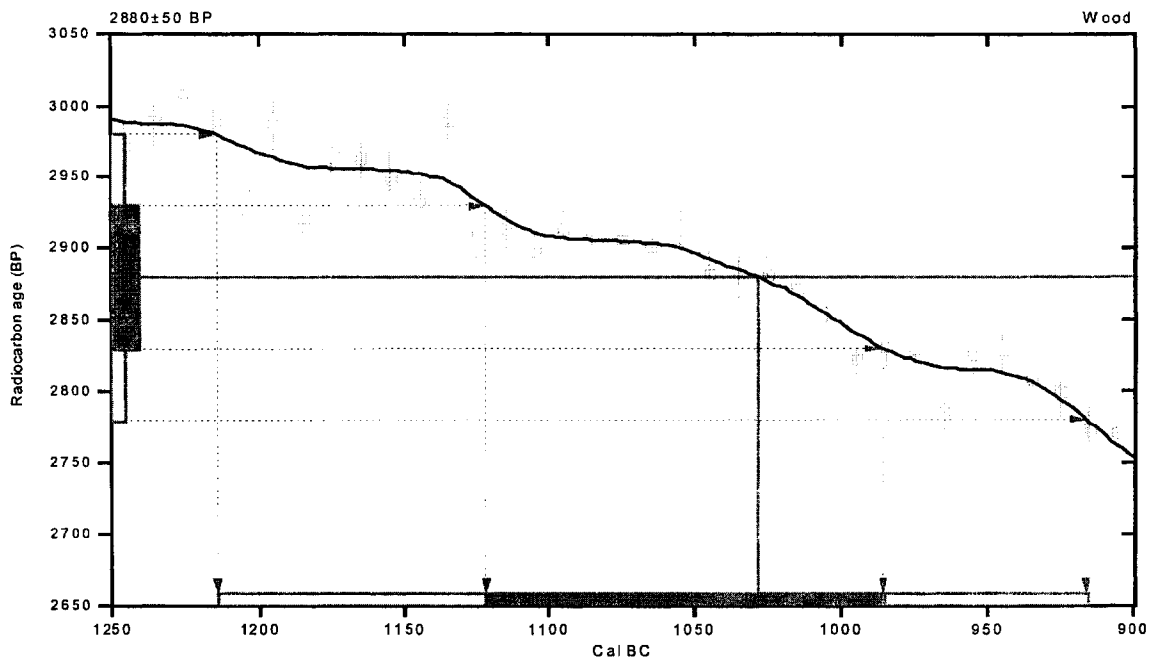
Conventional radiocarbon age: **2880±50 BP**

2 Sigma calibrated result: Cal BC 1210 to 920 (Cal BP 3160 to 2870)
(95% probability)

Intercept data

Intercept of radiocarbon age
with calibration curve: **Cal BC 1030 (Cal BP 2980)**

1 Sigma calibrated result: Cal BC 1120 to 990 (Cal BP 3070 to 2940)
(68% probability)



References:

Database used

Calibration Database

Editorial Comment

Stuiver, M., van der Plicht, H., 1998, *Radiocarbon* 40(3), pxii-xiii

INTCAL98 Radiocarbon Age Calibration

Stuiver, M., et. al., 1998, *Radiocarbon* 40(3), p1041-1083

Mathematics

A Simplified Approach to Calibrating C14 Dates

Talma, A. S., Vogel, J. C., 1993, *Radiocarbon* 35(2), p317-322

Beta Analytic Inc.

4985 SW 74 Court, Miami, Florida 33155 USA • Tel: (305) 667 5167 • Fax: (305) 663 0964 • E-Mail: beta@radiocarbon.com

Appendix B:
Figures and Tables from Lund (1996)

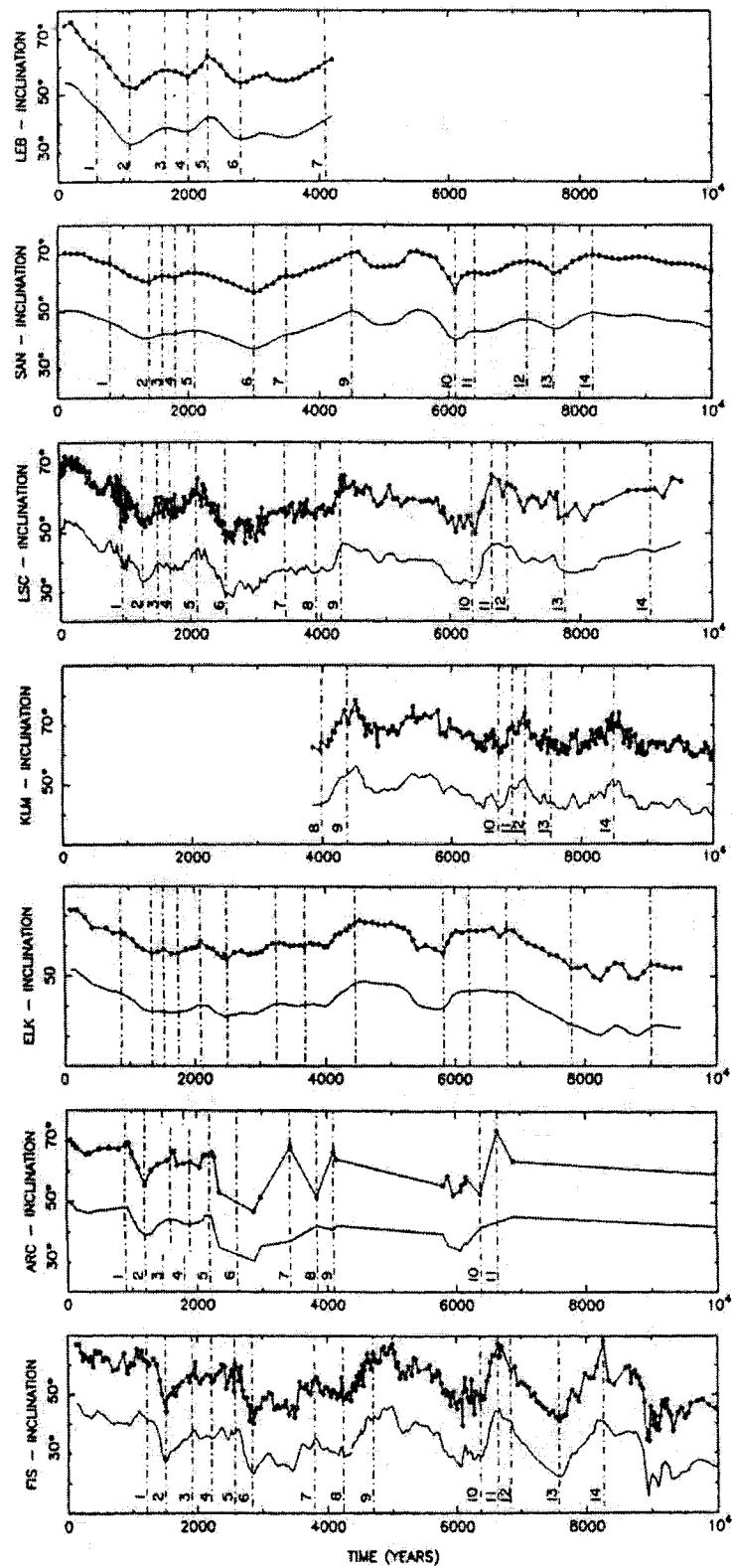


Figure from Lund (1996), page 8009.

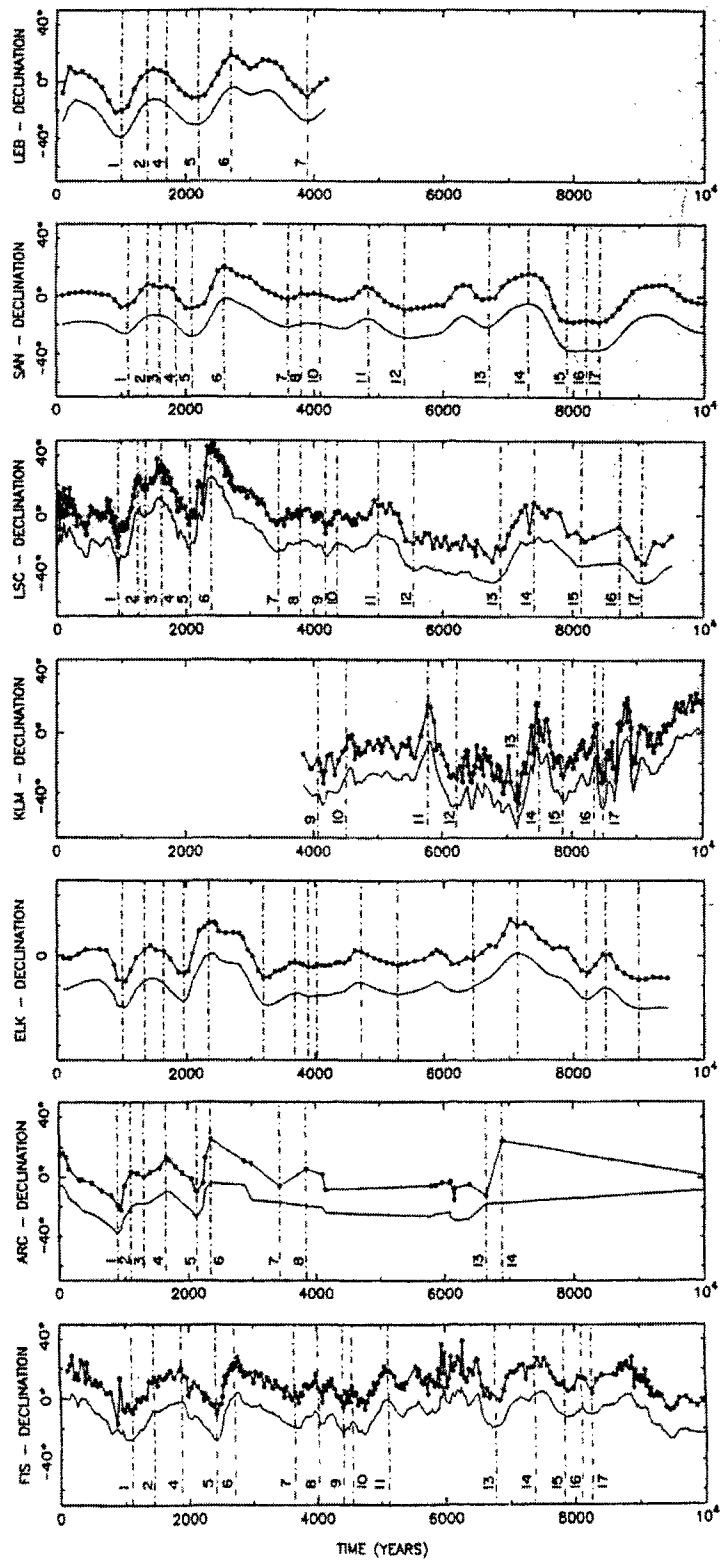


Figure from Lund (1996), page 8010.

Table from Lund (1996), page 8013

Feature*	FIS + Age, radio- carbon years	ARCMAG+ Age, radio- carbon years	BLU+ Age, radio- carbon years	ELK+ Age, radio- carbon years	KLM+ Age, radio- carbon years	LSC+ Age, radio- carbon years	SAN+ Age, radio- carbon years	LEB+ Age, radio- carbon years	SEN+ Age, radio- carbon years	Average Age, radio- carbon years
I1	(1190±90)†	900±100	(630± 80)	870±120	---	960± 90	750±150	(600±100)	---	870±180
I2	(1525±105)	1200±100	(910± 80)	1340±100	---	1310±140	1400±100	1200±100	1050±150	1250±180
I3	(1915± 65)	1475± 75	(1110±150)	1525±100	---	1520± 40	1600±100	1650±150	1550±150	1555±120
I4	(2280± 80)	1875±175	(1420±160)	1745±100	---	1675± 85	1800±100	2000±100	1950±150	1840±210
I5	(2585± 85)	2190±80	(1670± 80)	2090±190	---	2100± 70	2100±100	2350±150	2300±200	2190±230
I6	2870± 90	2615±90	2630± 80	2500±110	---	2530±130	3000±100	2850±150	2900±200	2735±380
I7	(3715±145)	3440§	---	3250±350	---	3425±145	3550±150	(4100±100)	(3850±150)	3415±250
I8	(4200±180)	3850§	---	3690± 90	3985±145	3915±195	---	---	(4400±200)	3860±250
I9	4635±105	(4100§)	---	4455±100	4350±180	4385±135	4550±150	---	(5000±200)	4475±240
I10	6300±130	6390±120	---	(5820± 70)	(6745±105)	6315± 95	6100±100	---	6050±150	6230±300
I11	6685±155	6640±130	---	(6225±220)	(6920± 50)	6710± 60	6400±100	---	6350±150	6555±340
I12	6855±155	---	---	6805±110	7110± 60	6955± 65	7200±100	---	7000±200	6990±300
I13	7530±150	---	---	7795±210	7540± 90	7740±330	7600±100	---	7800±200	7665±250
I14	8225±115	---	---	(9005±200)	8485± 85	(9200±200)	8200±100	---	8700±200	8400±470
D1	1045±175	800±150	(630± 80)	1000±100	---	955± 75	1050±150	950±150	(700±200)	965±180
D2	1455± 85	1100±100	(850±150)	1340±100	---	1225± 55	1400±100	1400±100	1150±150	1295±270
D3	---	1300±100	---	---	---	1365± 55	(1600±100)	---	---	1330± 90
D4	(1910± 60)	1675±125	(1420±150)	1645±110	---	1640± 80	1700±100	1700±100	(1350±150)	1670± 60
D5	(2415± 85)	2140± 90	(1740± 80)	1960± 80	---	2065±105	2100±100	2150±150	1950±150	2060±180
D6	2740±100	2385±115	2410± 80	2350±110	---	2460±140	2600±100	2700±100	2600±100	2530±300
D7	3670±100	3440§	3320± 80	3200±100	---	3415±135	3600±100	(3900±100)	3400±200	3435±320
D8	(4040±240)	3850§	---	3690±140	---	3800±220	3800±100	---	(4050±150)	3785±140
D9	(4365± 65)	---	---	3895± 80	4015±175	4165±145	---	---	---	4025±270
D10	4570± 40	---	---	4030± 80	4535± 85	4415±105	4100±100	---	---	4330±500
D11	5105±105	---	---	4720±100	(5760±100)	5070±120	4800±100	---	(5400±200)	4925±380
D12	---	---	---	5290±210	(6190±130)	5560±120	5400±100	---	5700±200	5485±360
D13	6730±170	6570±190	---	6450±100	(7165±115)	6860± 90	6700±100	---	6450±150	6625±330
D14	7320±310	(6890§)	---	7135±110	7485±125	7510±300	7300±100	---	7200±200	7325±300
D15	7855±125	---	---	8200±150	7870±150	8140±200	8000±100	---	8300±200	8060±360
D16	8095± 45	---	---	8500±100	8340± 40	(8705±165)	8200±100	---	---	8280±350
D17	8250± 90	---	---	(9005±150)	8480± 50	(9130±130)	8400±100	---	---	8375±230

*I1 through I14 are inclination features; D1 through D17 are declination features.

+FIS, Fish Lake; ARCMAG, composite of archeomagnetic and lava flow data; BLU, Blue Lake; ELK, Elk Lake; KLM, Kylene Lake; LSC, Lake St. Croix; SAN, Sandy Lake; LEB, Lake LeBeouf; SEN, Seneca Lake.

§Radiocarbon ages in parentheses were not used to estimate the final Average age for each PSV feature.

§Age of vector indicative of correlated feature. ARCMAG does not contain enough data in this time interval to define a time interval for the feature.

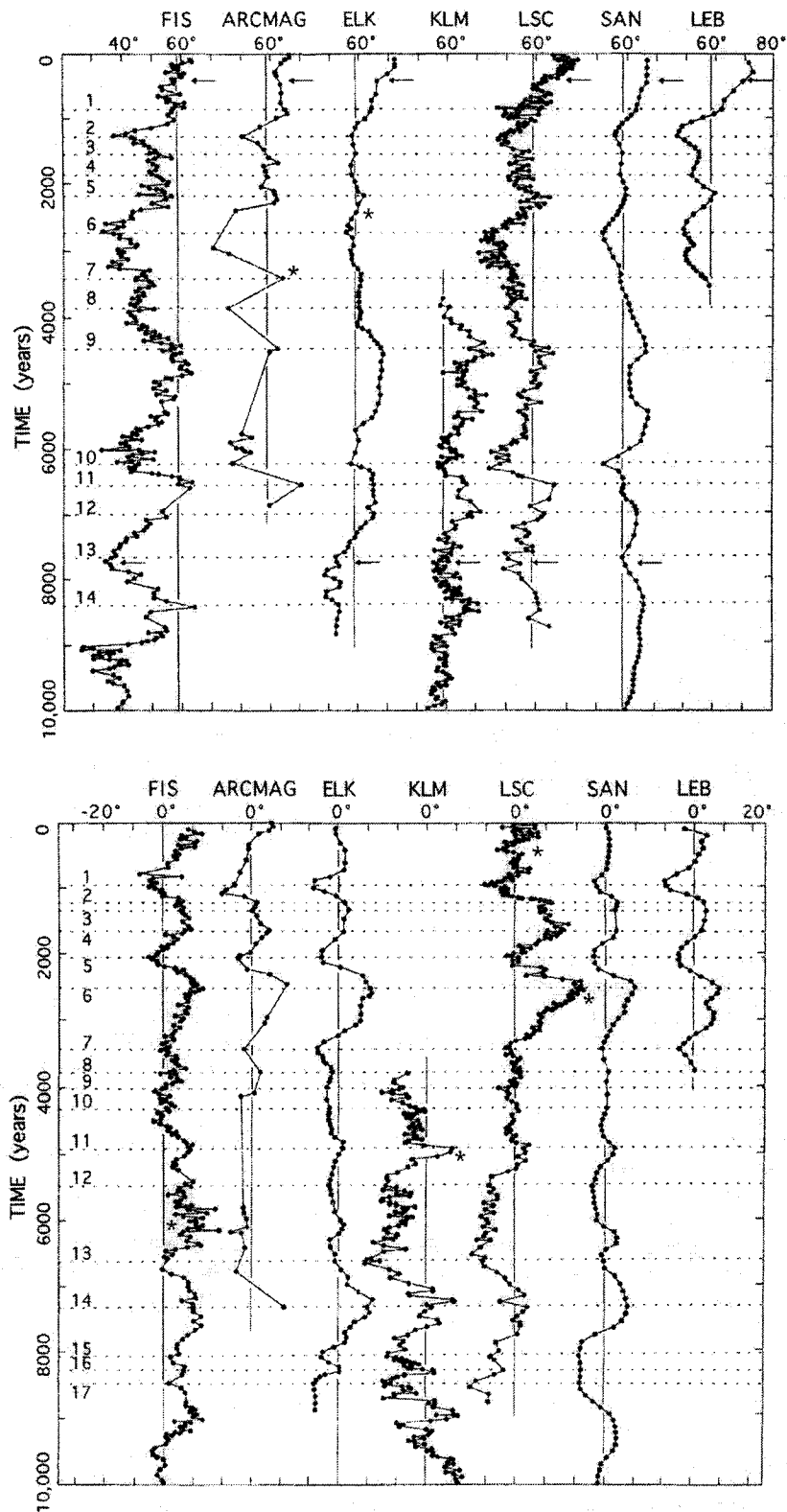


Figure from Lund (1996), page 8016.

Appendix C

Core Description Sheets



LRC
INITIAL CORE DESCRIPTION

Lake Flathead Lk, MT
CORE ID FL-00-9P-III
Date 8/12/02
Observer MSH

SECTION LENGTH 88.7
SED. LENGTH 88.5
CUM. SED LENGTH _____

mbf top _____
mbf bot _____

cum. mbf	Disturb.	COLOR	Struc.	UNIT	LITHOLOGIC DESCRIPTION	SAMPLES
10						10-10
20						20-20
30						30-30
40						40-40
43.5						43.5
45.5						45.5 (ash)
50						50-50
60						60-60
61						61 (ash)
70						70-70
72.5						72.5 (ash)
74.5						74.5 (ash)
80						80-80
84						84
100						100
110						110
120						120
130						130
140						140
150						150

Note - Core split ~ Dec, 2000 in Missoula. Split surface is oxidized by the time of this description.



LRC
INITIAL CORE DESCRIPTION

Lake Flathead Lake, MT
CORE ID FL-00-9P-SI
Date 8/12/02
Observer MJH

SECTION LENGTH 150.1
SED. LENGTH 149.9
CUM. SED LENGTH _____

mbif top _____
mbif bot _____

Disturb.	COLOR	Struc.	UNIT	LITHOLOGIC DESCRIPTION	SAMPLES
10					10
20					20
30					30
40					40
50					50
60					60
70					70
80					80
90					90
100					100
110					110
120					120
130					130
140					140
150					150

core was split in Dec, 2004;
cut surface is oxidized at
the time of this description.
No sulfides or other evidence
of stratification is visible.

5/2 10/12

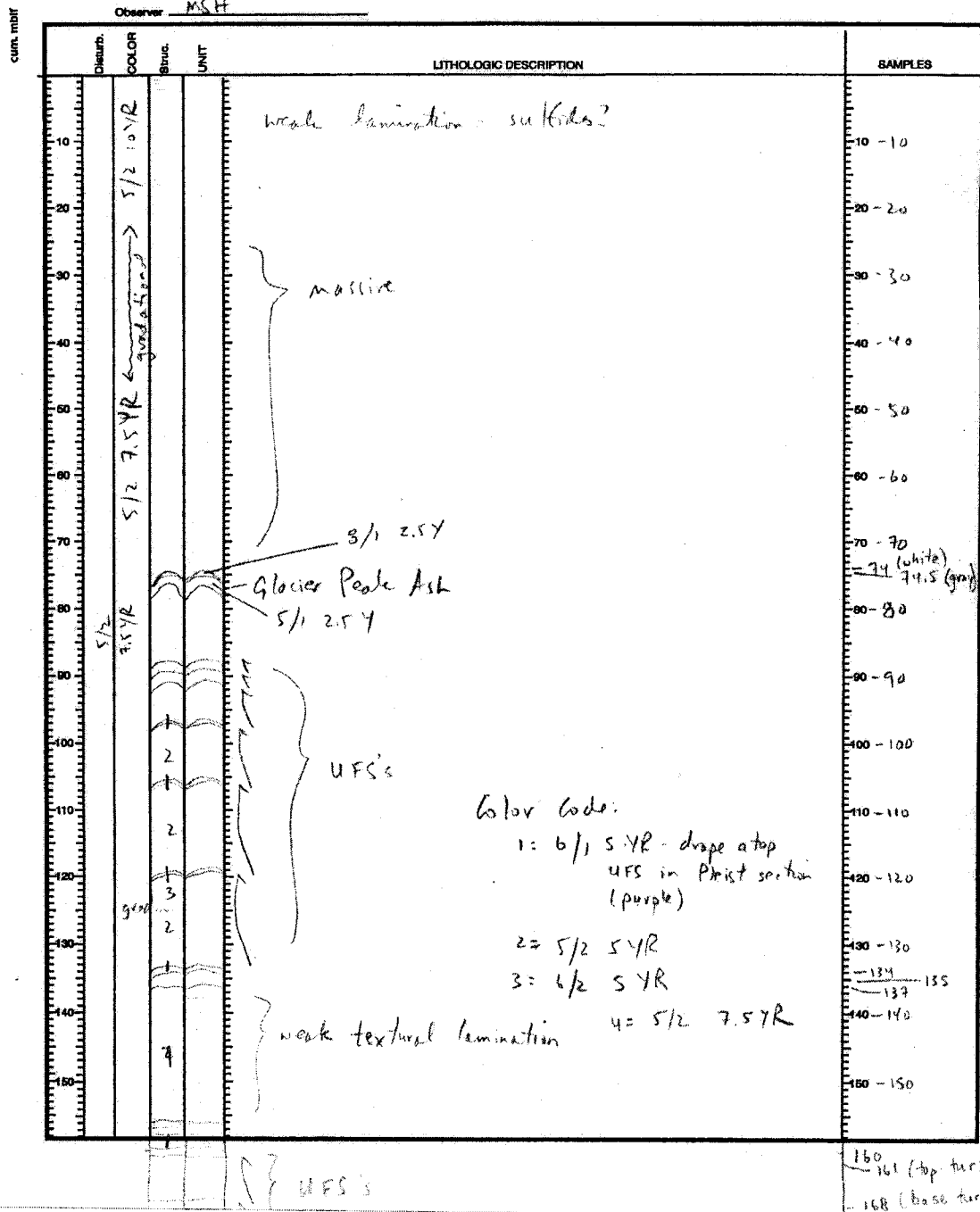


LRC
INITIAL CORE DESCRIPTION

Lake Flathead Lk, MT
CORE ID FL-00-9P-V
Date 8/12/02
Observer MSH

SECTION LENGTH 176.7
SED. LENGTH 176.2
CUM. SED LENGTH _____

mbif top _____
mbif bot _____



Appendix D:

Data Figures

In this appendix I have included figures of all data collected and/or calculated on the three cores discussed in this dissertation, FL-00-9G, FL-00-9P, and FL-02-9Gb. Figures are presented utilizing current dating models for the particular core. The digital data used to create the figures can all be found within Appendix F. Most of the data is contained in a master spreadsheet created for each core called '*(core name)*-Master-Calc.xls'. The files are located on Flathead CD #1, in a folder named 'Data Master' under each core data folder. Uncertainty at 2σ is show gray bars.

FL-00-9G Data

Figure 59. Carbon/Nitrogen Data	220
Figure 60. Dating Model	221
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FL-00-9P Data

Figure 63. Carbon/Nitrogen Data	231
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Figure 67. Gray Scale Data	235
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FL-02-9Gb Data

Figure 72. Carbon/Nitrogen Data	249
Figure 73. Dating Model	250
Figure 74. Grain Size / %Water Data	252
Figure 75. Elemental Data	253

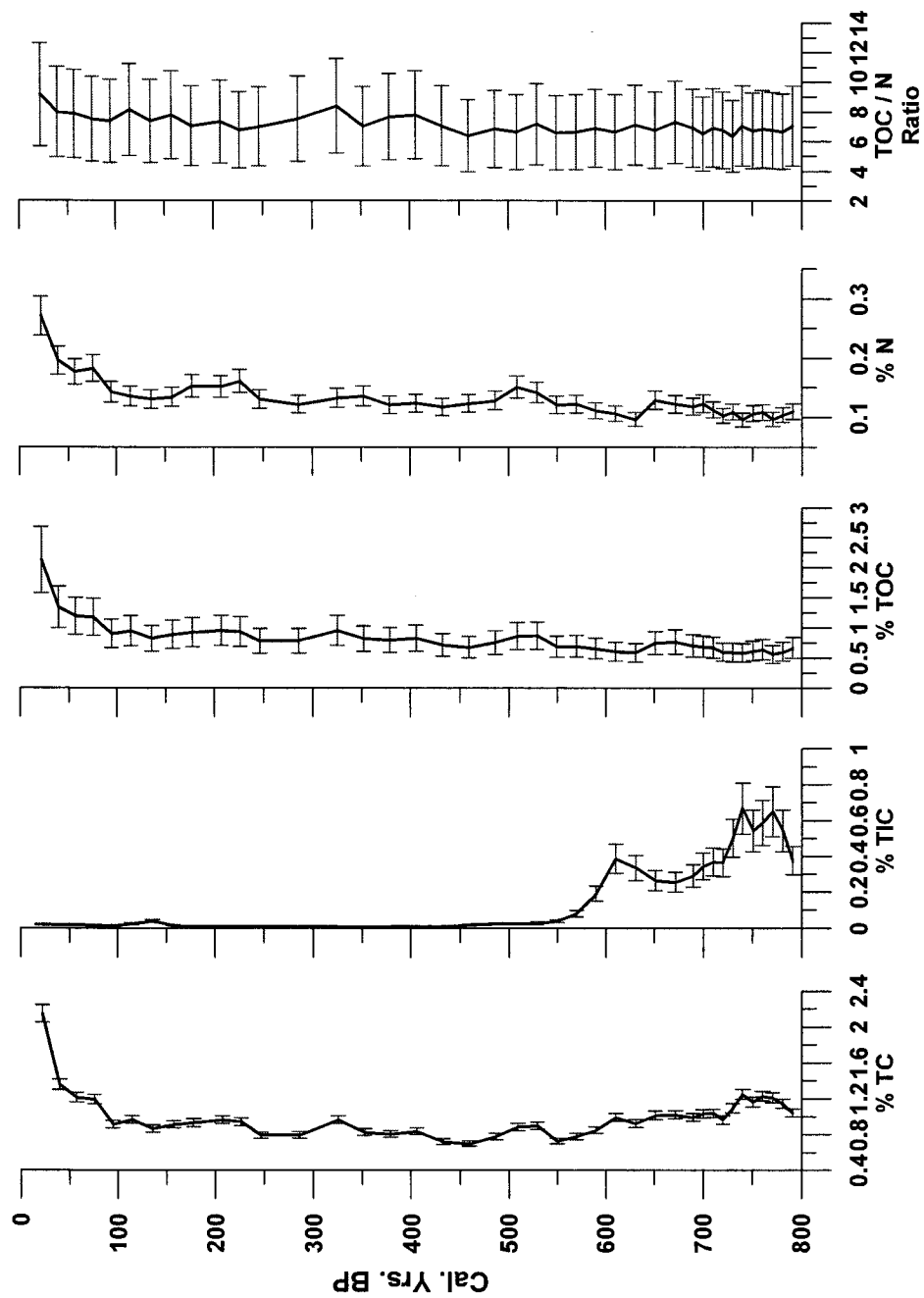


Figure 59 – Core FL-00-9G carbon and nitrogen data.

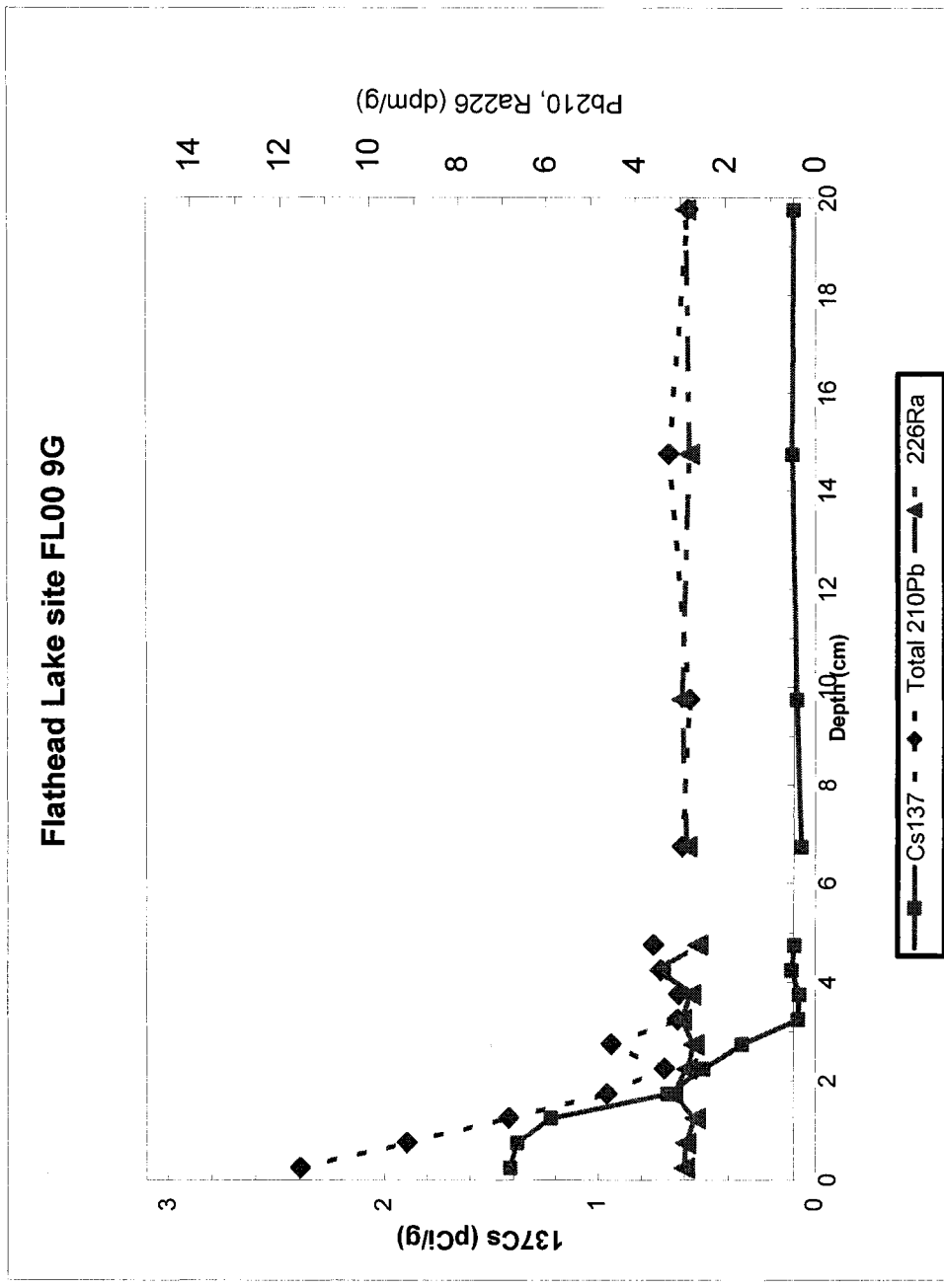


Figure 60 – Core FL-00-9G Radiometric Dating Data.

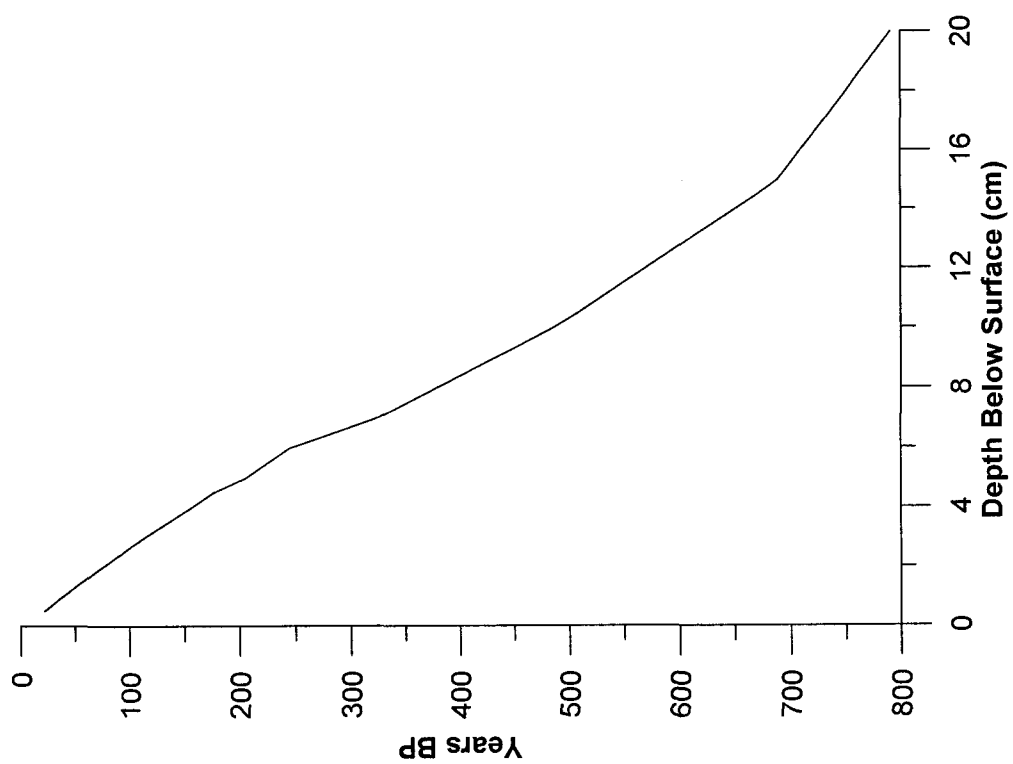


Figure 60 (con't) – Core FL-00-9G Dating Model.

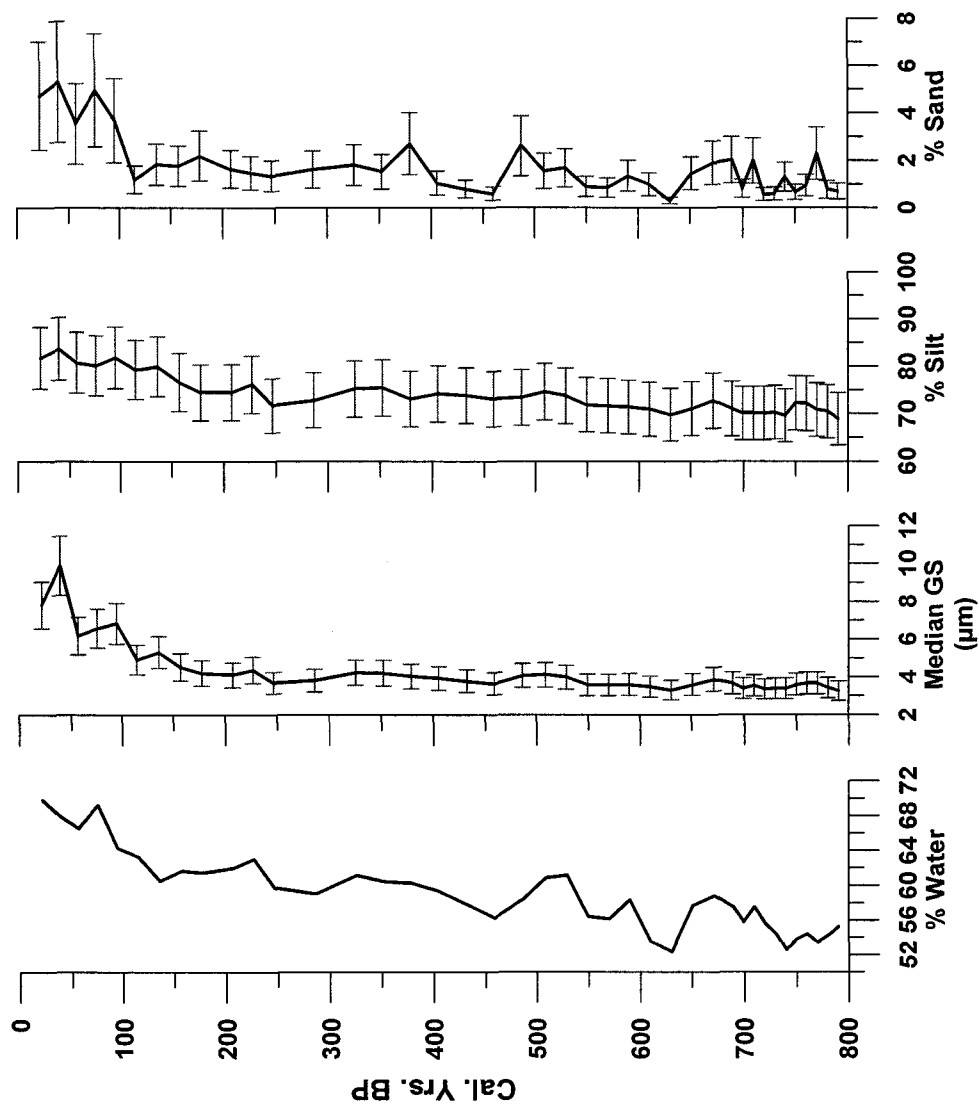


Figure 61 – Core FL-00-9G Water and Grain Size Data.

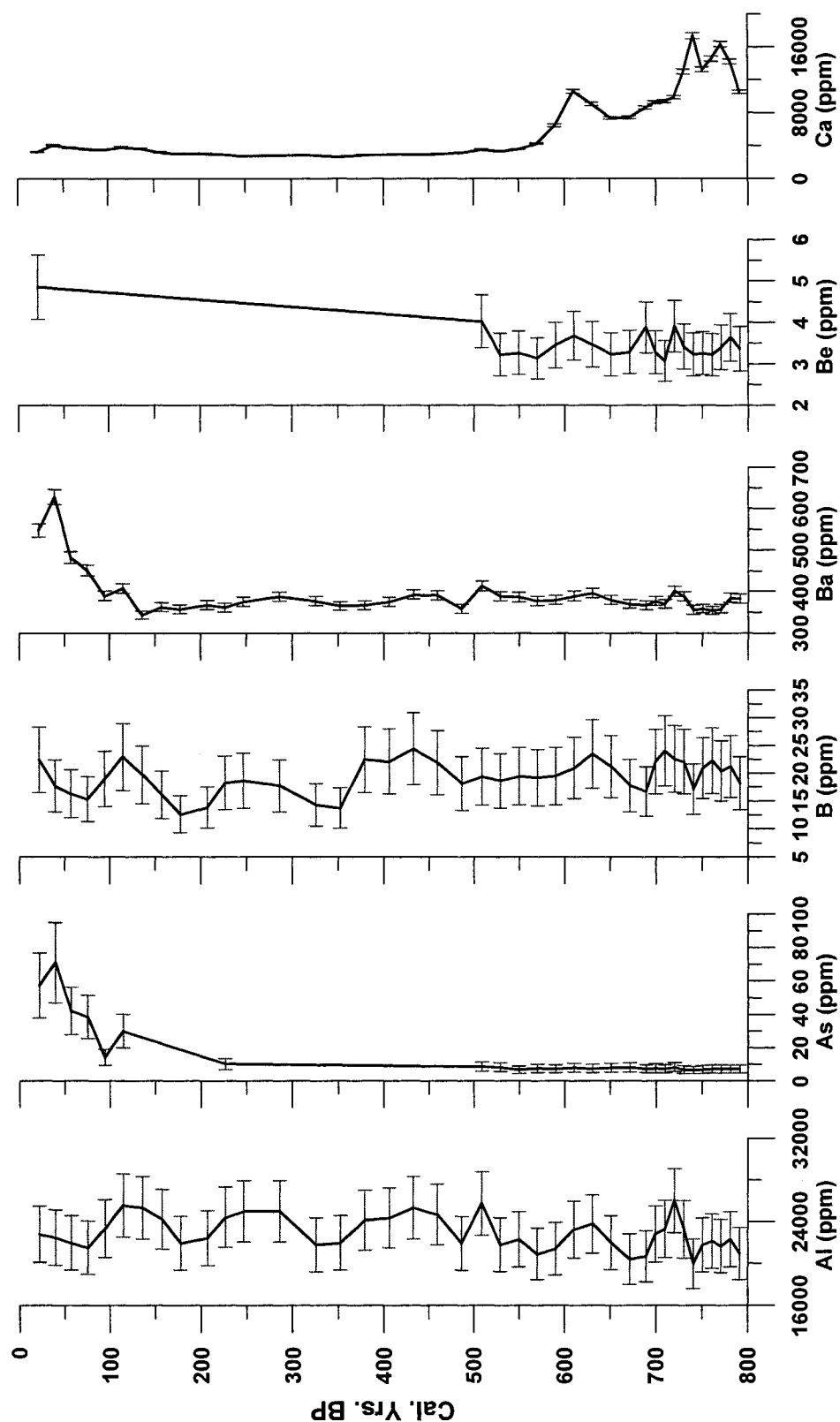


Figure 62 – Core FL-00-9G Elemental Data.

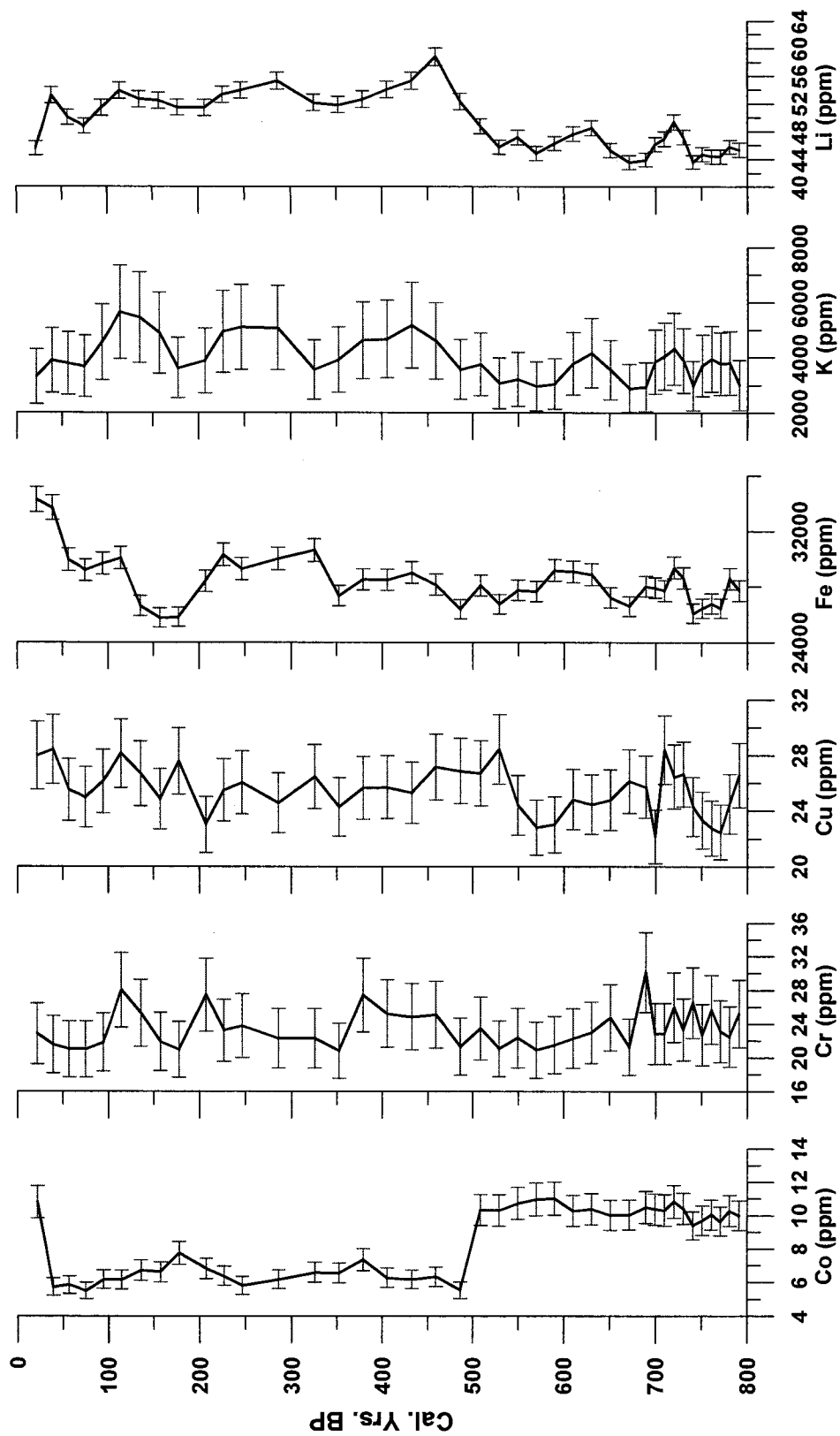


Figure 62 – Core FL-00-9G Elemental Data (con't).

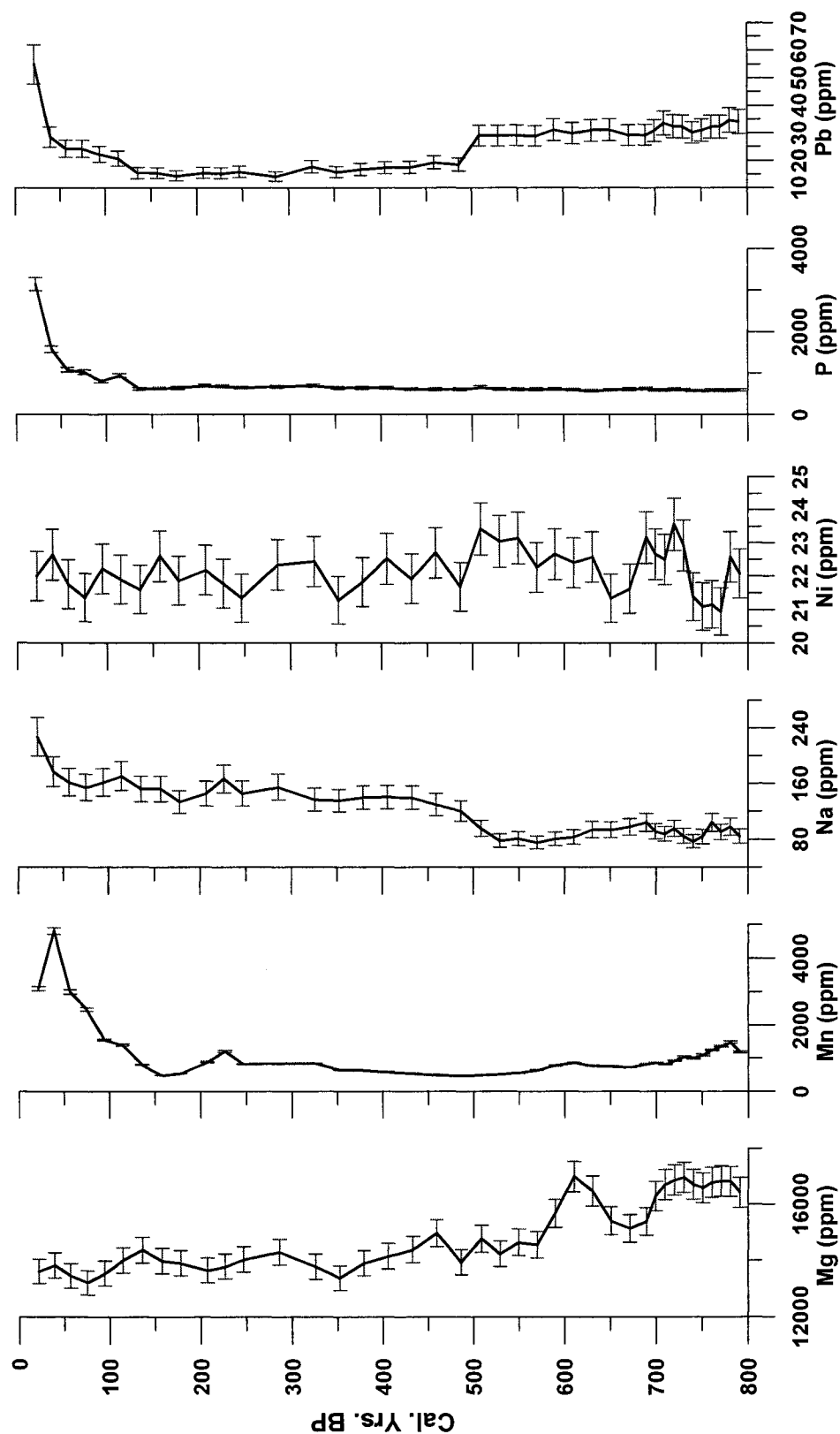


Figure 62 – Core FL-00-9G Elemental Data (con't).

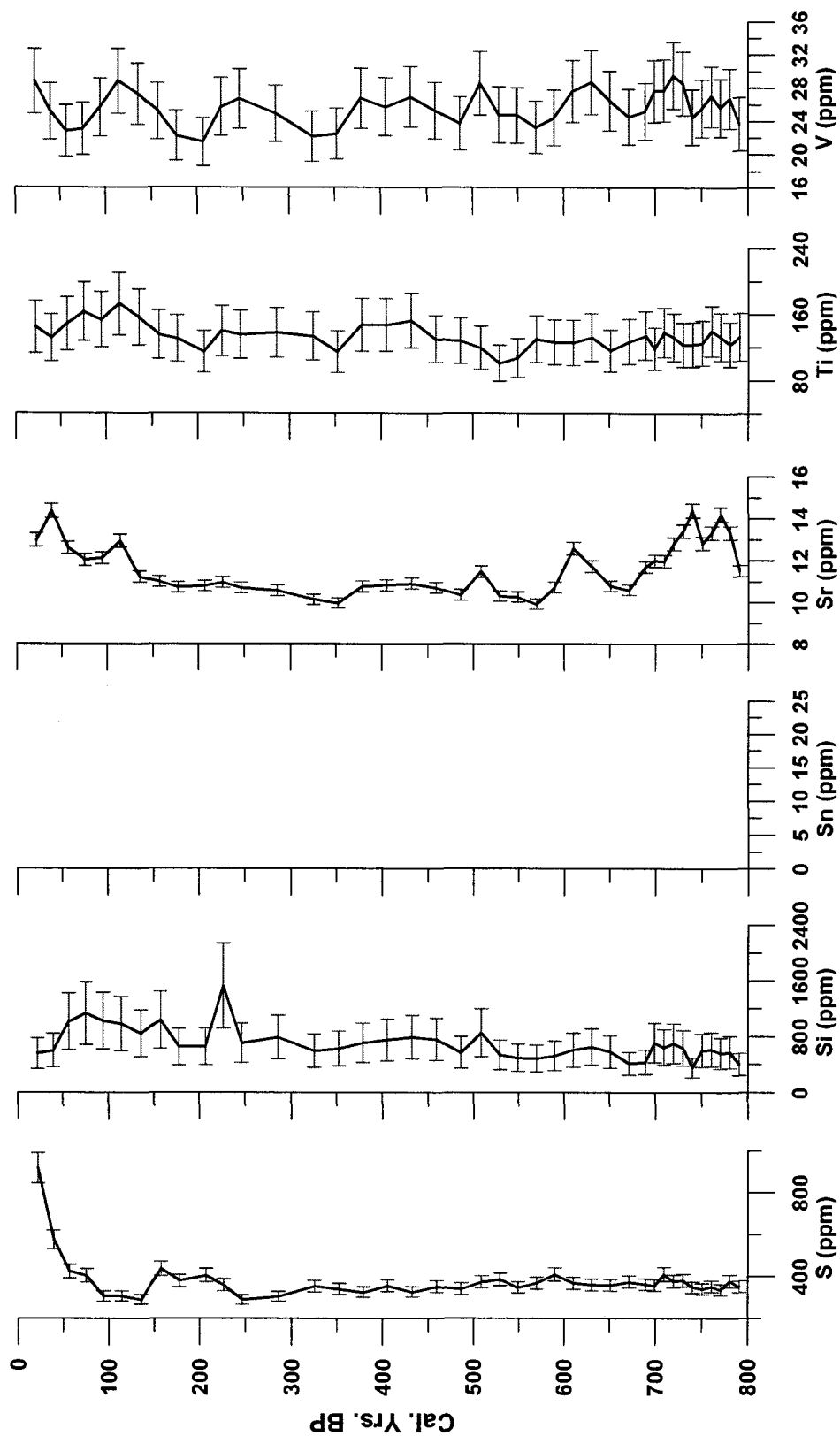


Figure 62 – Core FL-00-9G Elemental Data (con't).

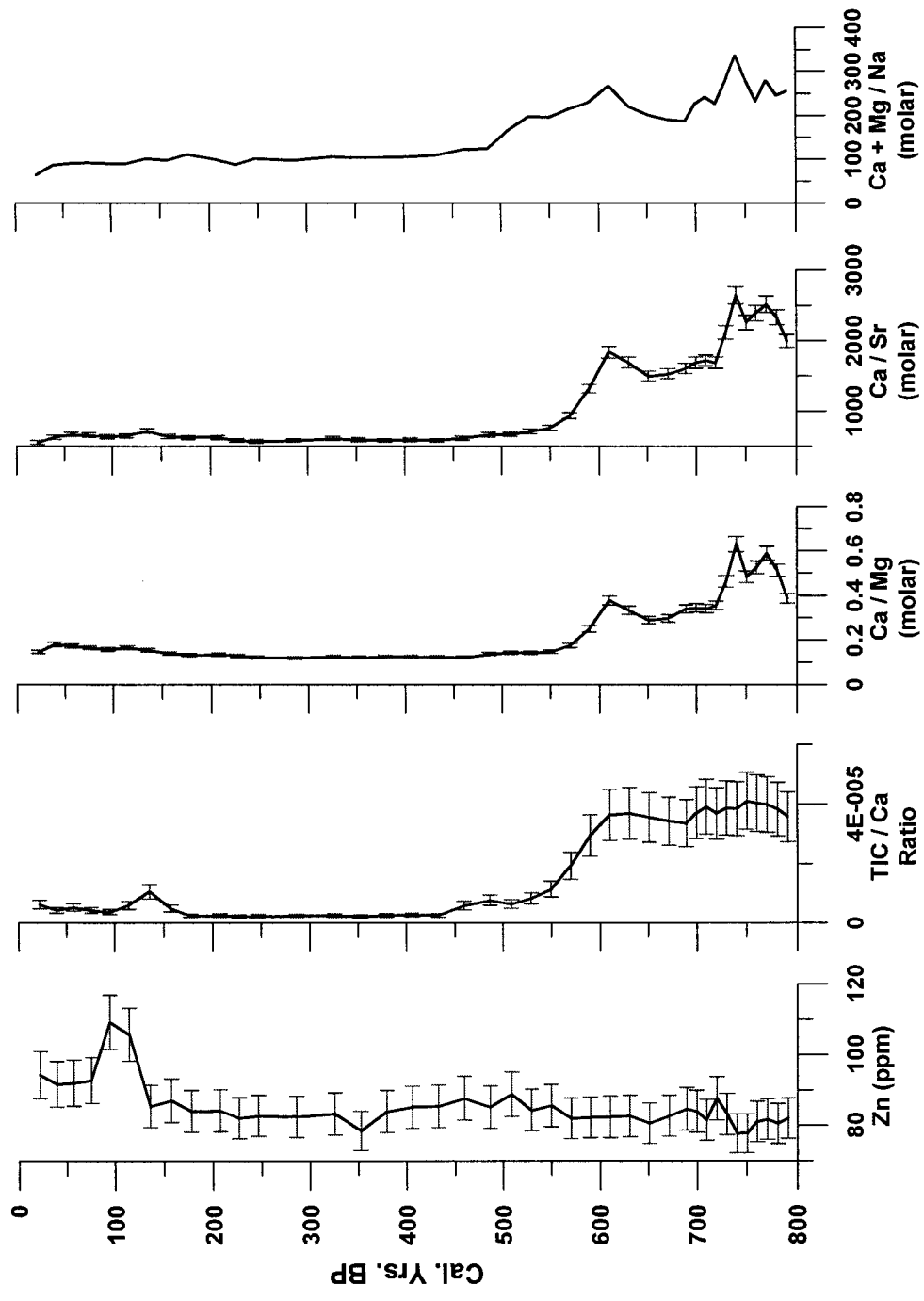


Figure 62 – Core FL-00-9G Elemental Data (con't).

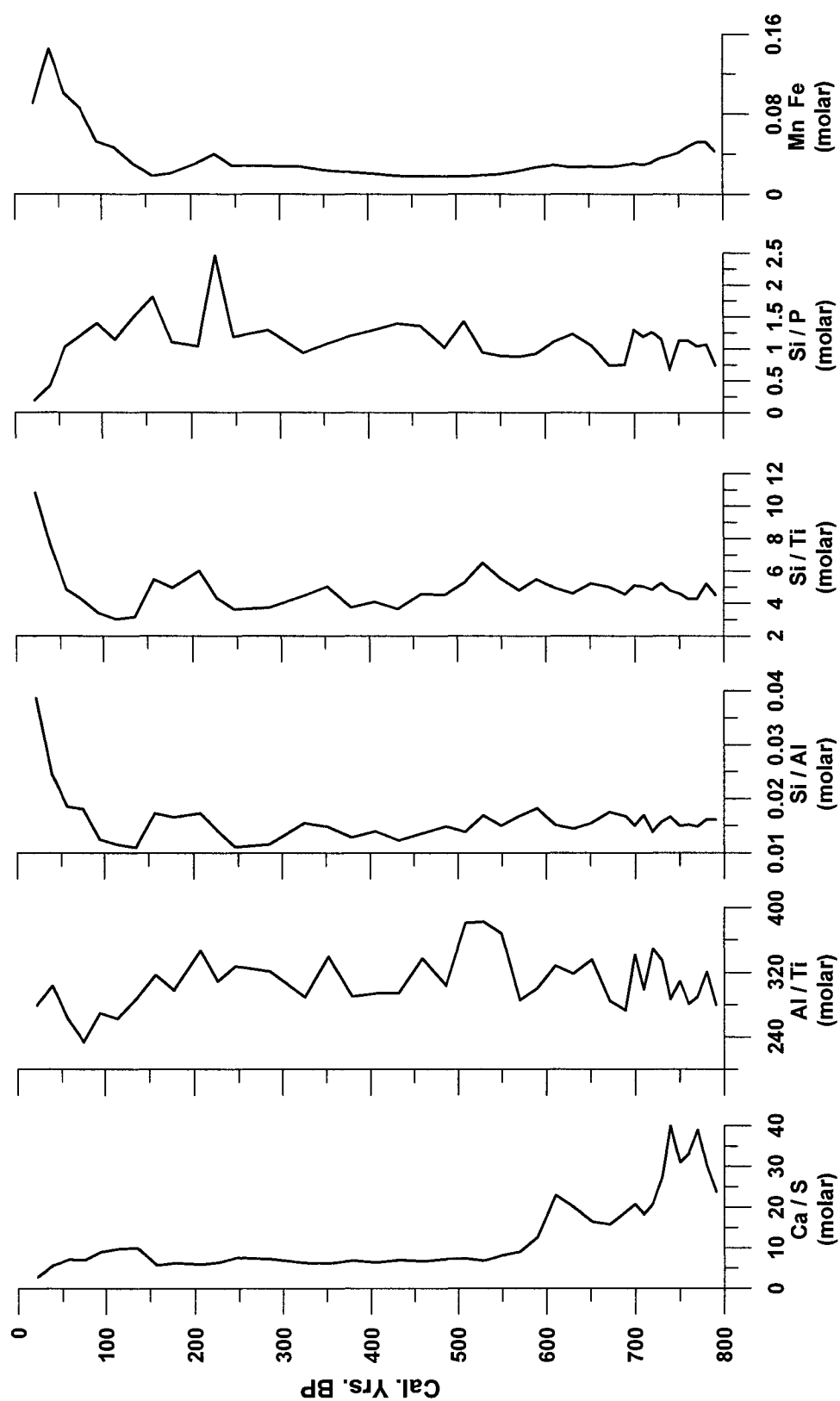


Figure 62 – Core FL-00-9G Elemental Data (con't).

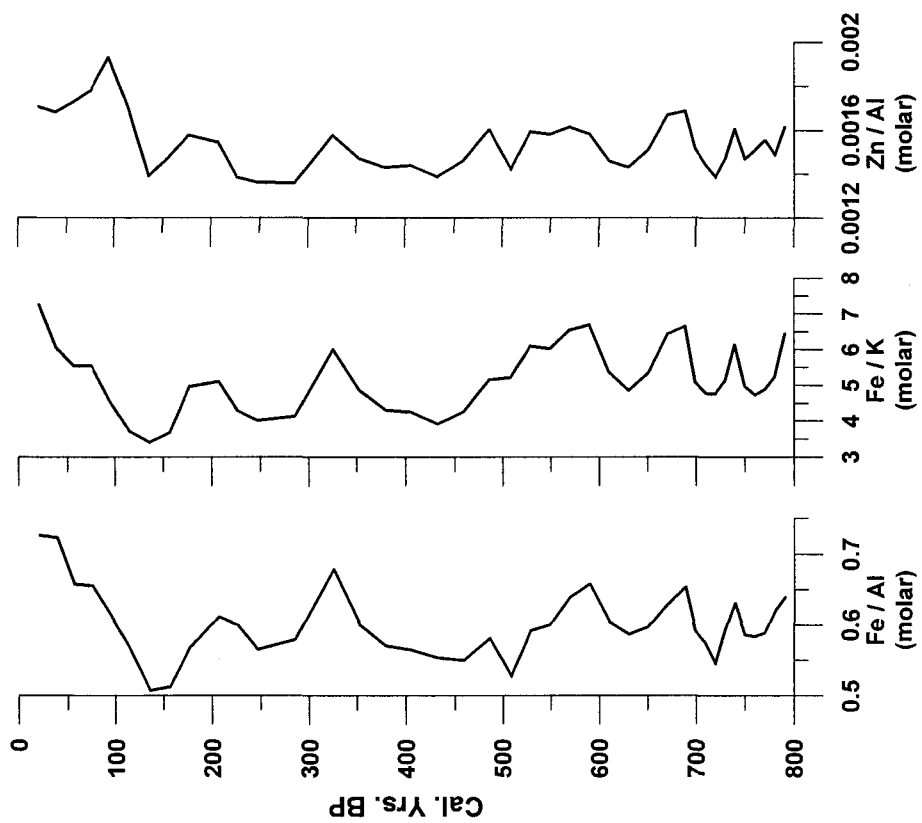


Figure 62 – Core FL-00-9G Elemental Data (con't).

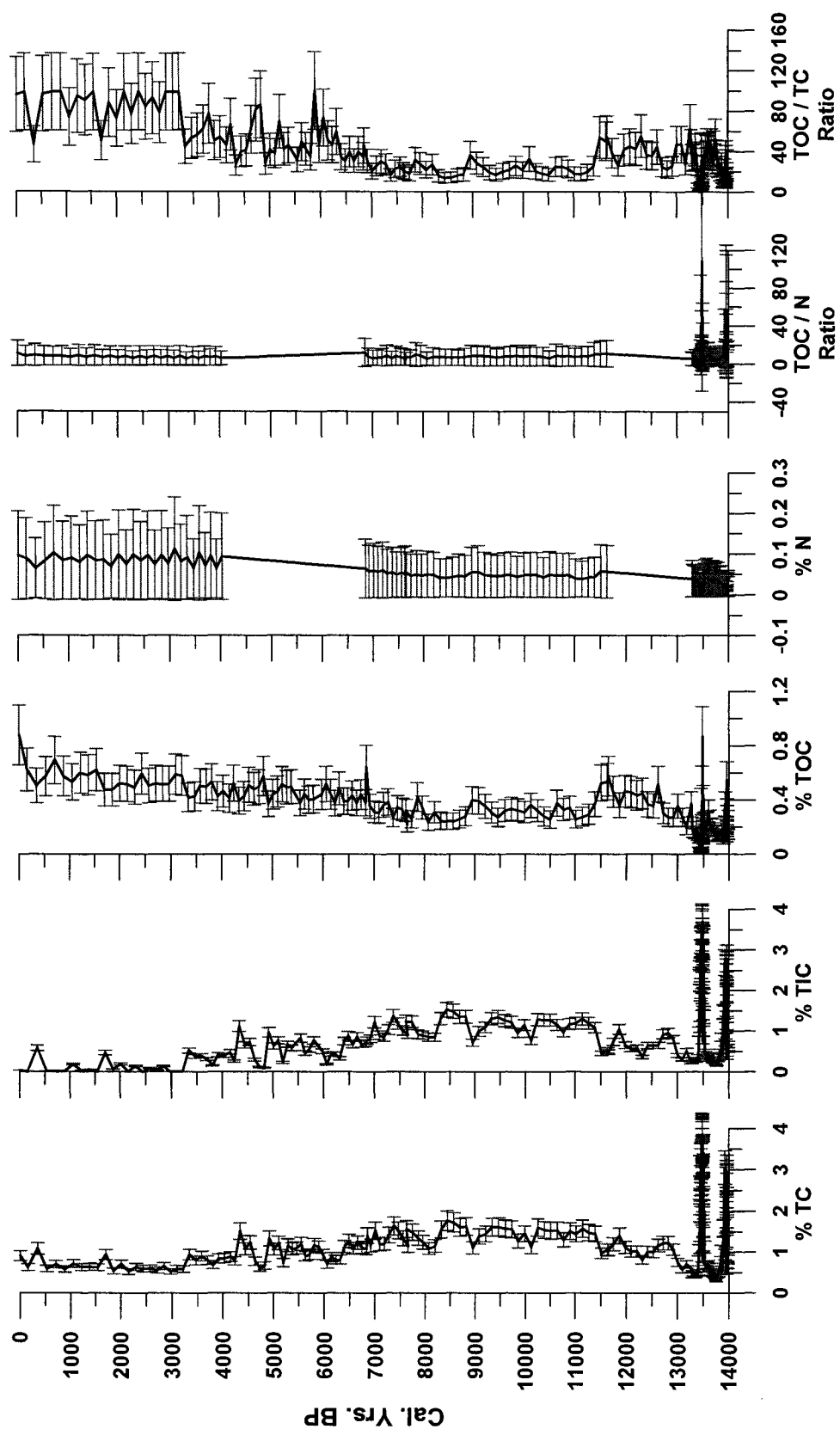


Figure 63 – Core FL-00-9P carbon and nitrogen data.

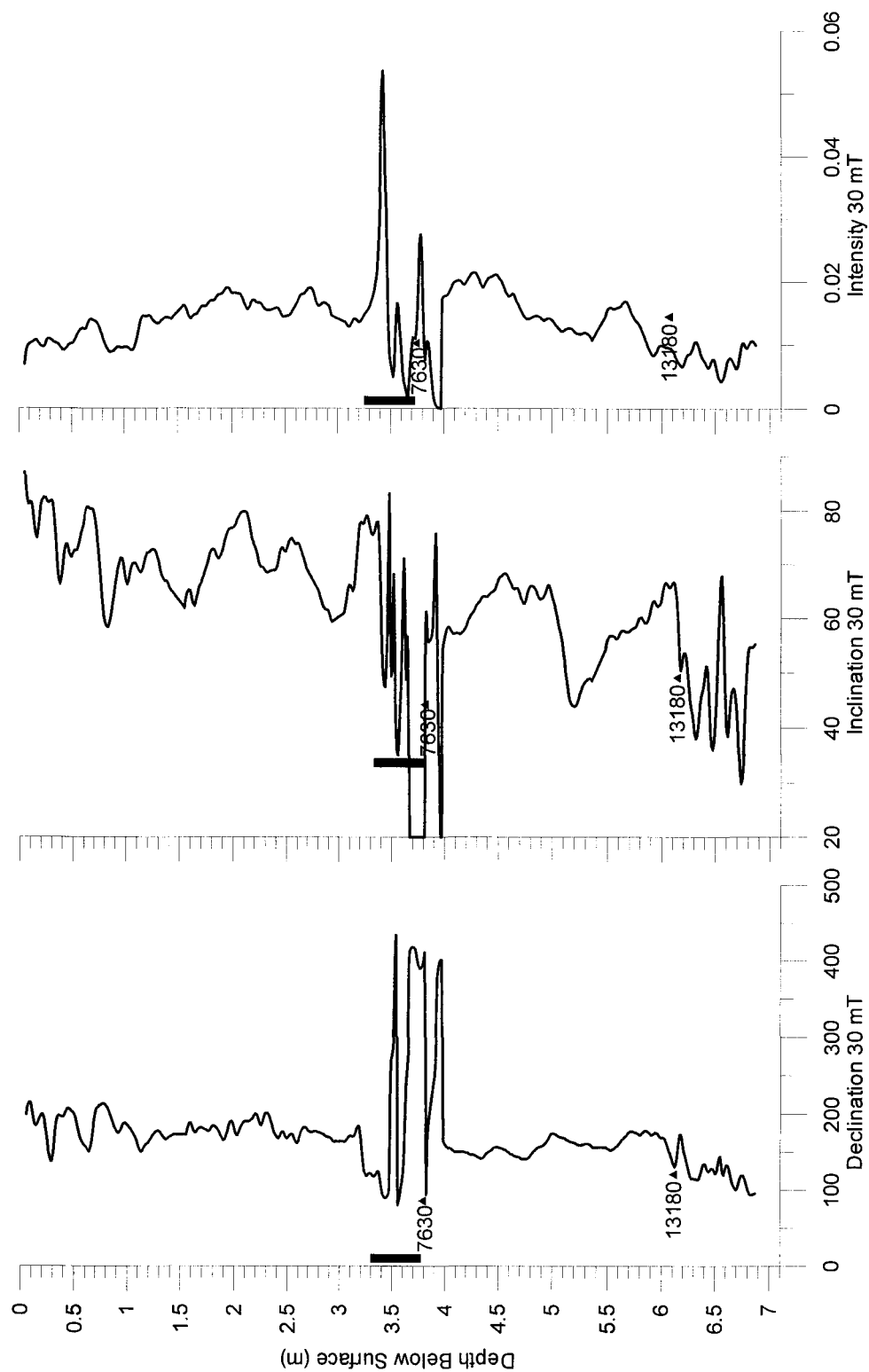


Figure 64 – Core FL-00-9P paleomagnetic secular variation data. Date points (in cal. yrs. BP) are the tephras found in core 9P, with the bar representing the thickness of the Mazama ash..

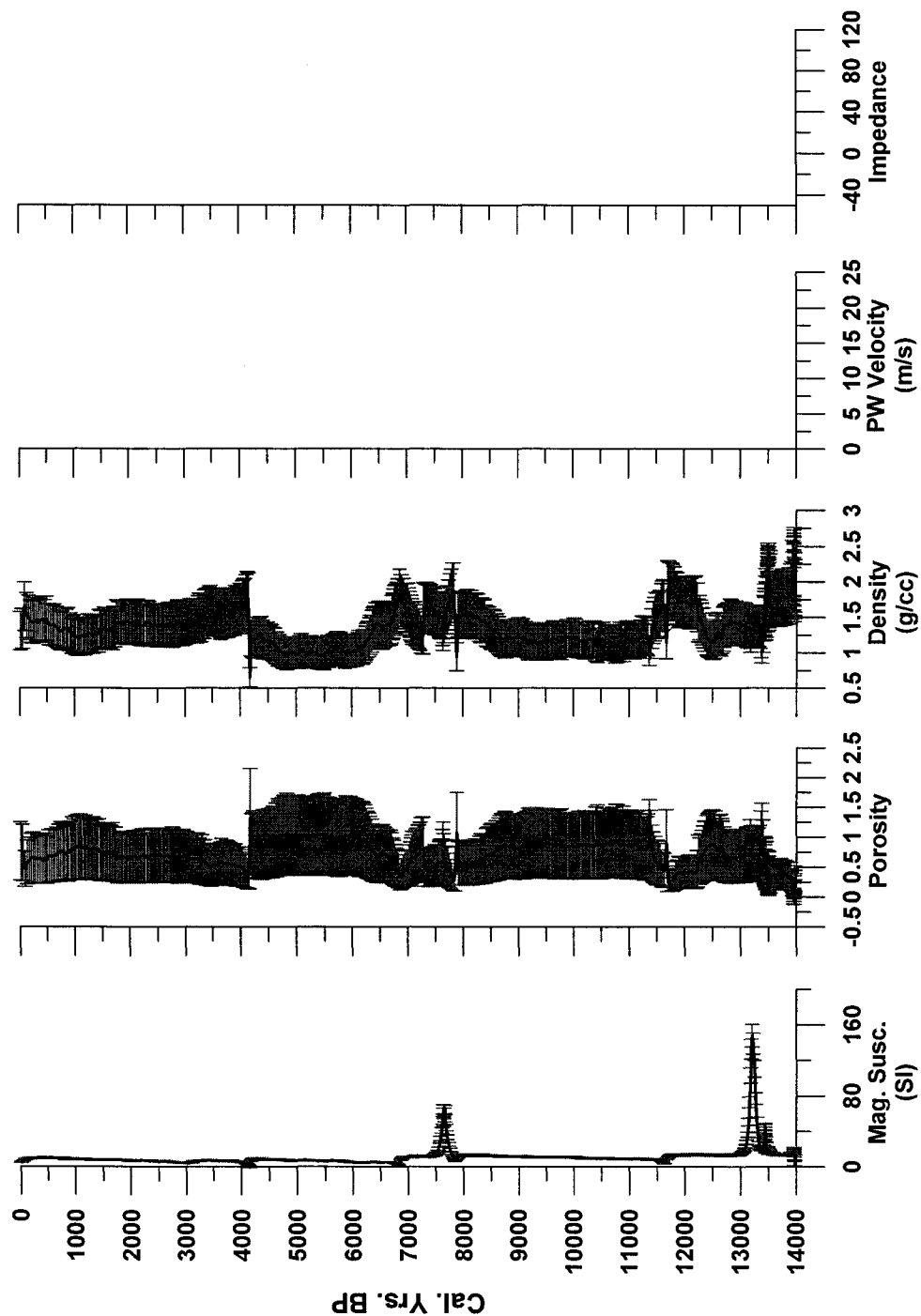


Figure 65 – Suite of Geotek analyses run on archived half of the split core sections. Data for velocity and impedance could not be collected on the split cores.

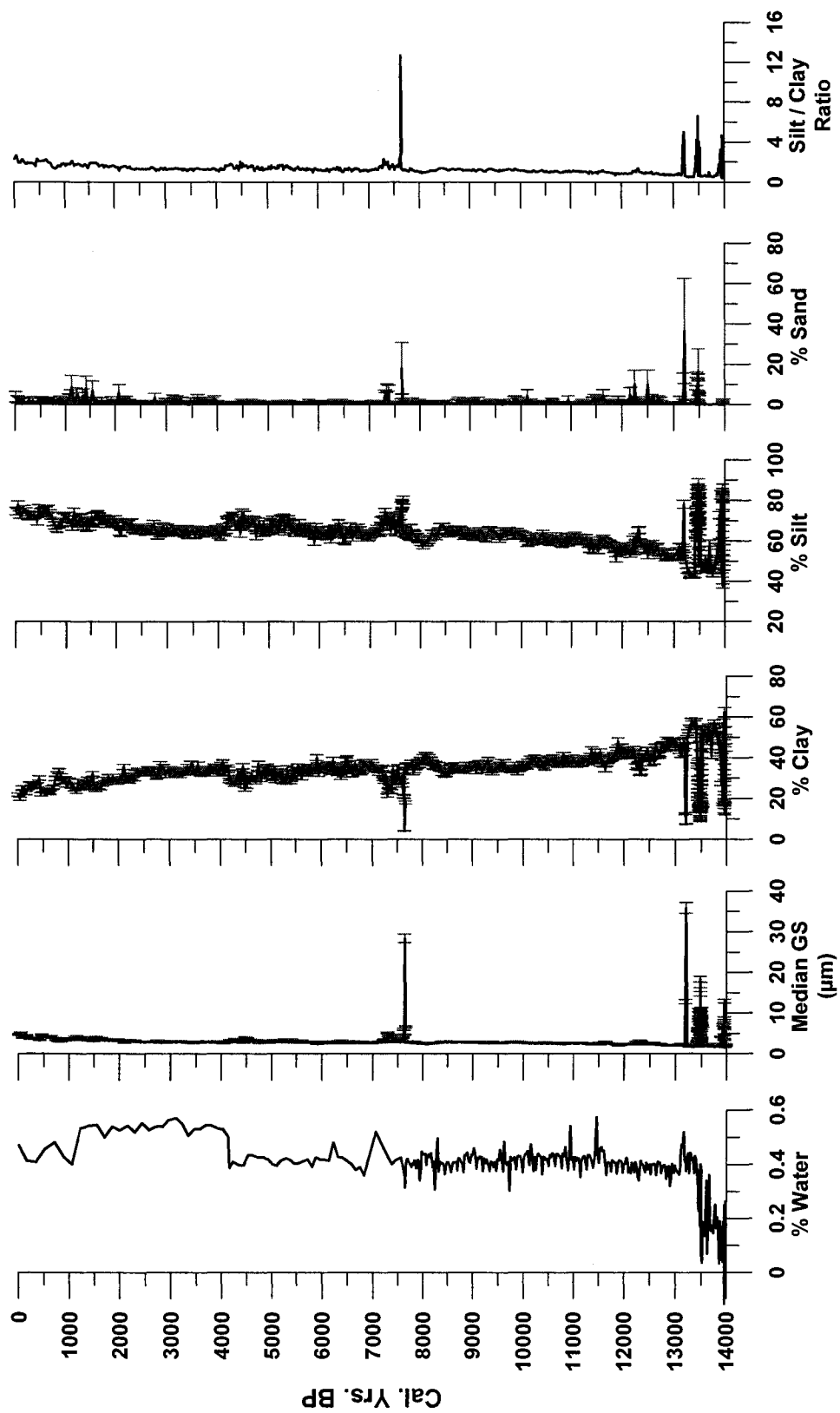


Figure 66 – Core FL-00-9P percent water content and grain size data.

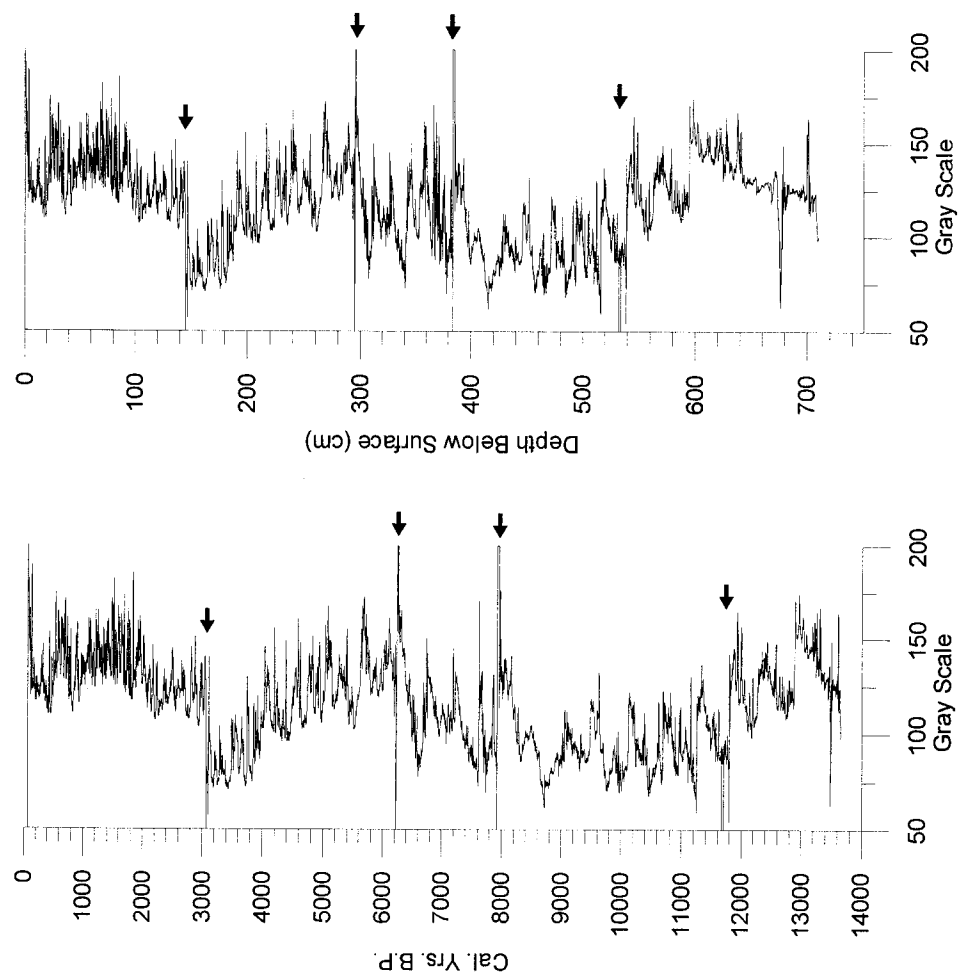


Figure 67 – Core FL-00-9P gray scale data. Figure shows gray scale measurements obtained from the Scion Imaging software based both on age and depth. Arrows point to breaks between core sections.

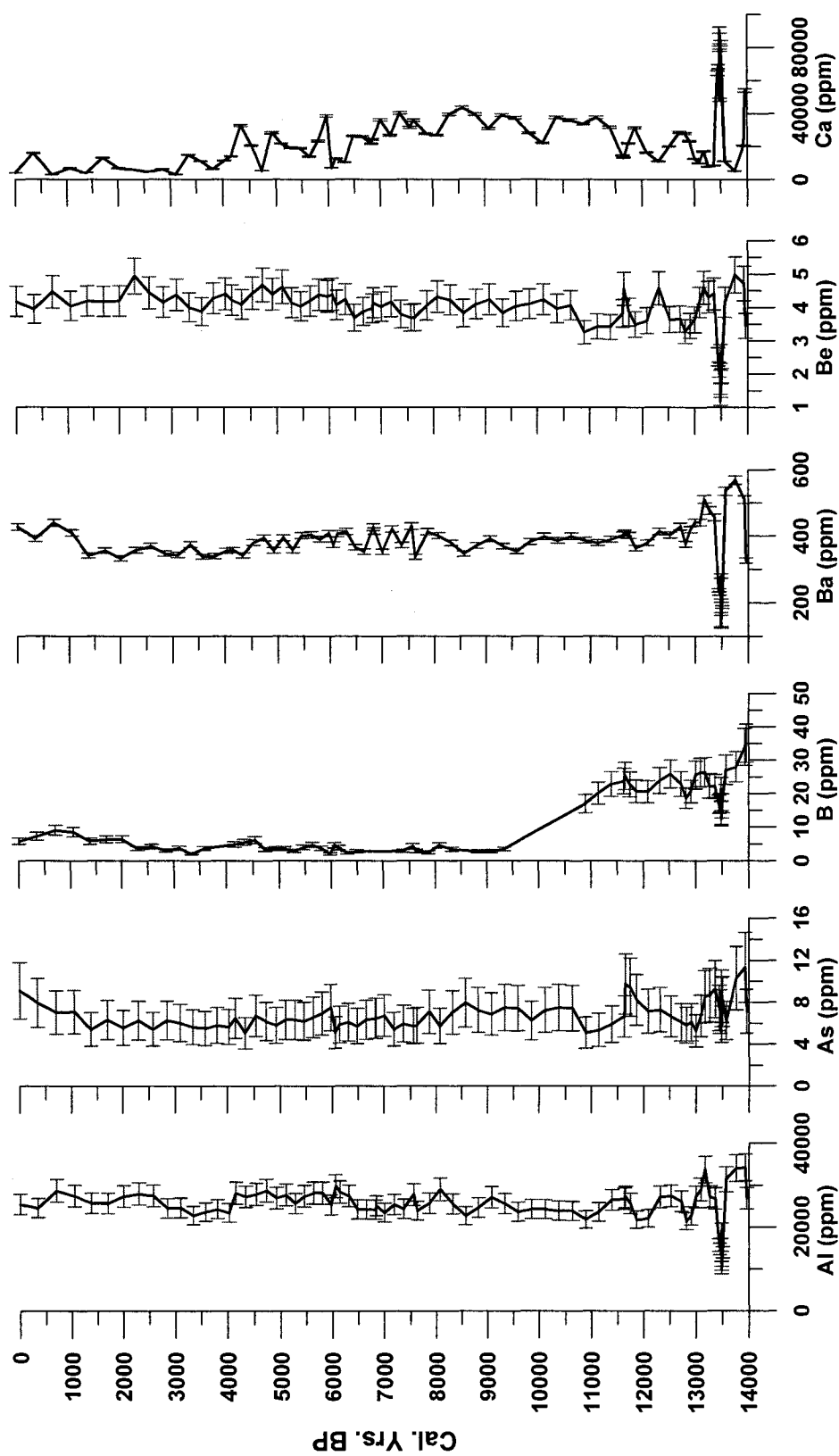


Figure 68 – Elemental data for core FL-00-9P. Portions of some elements were below detection limits of the ICP, these portions appear without error bars. Additionally, elements Cd, Hg, and Mo were completely below detection limits.

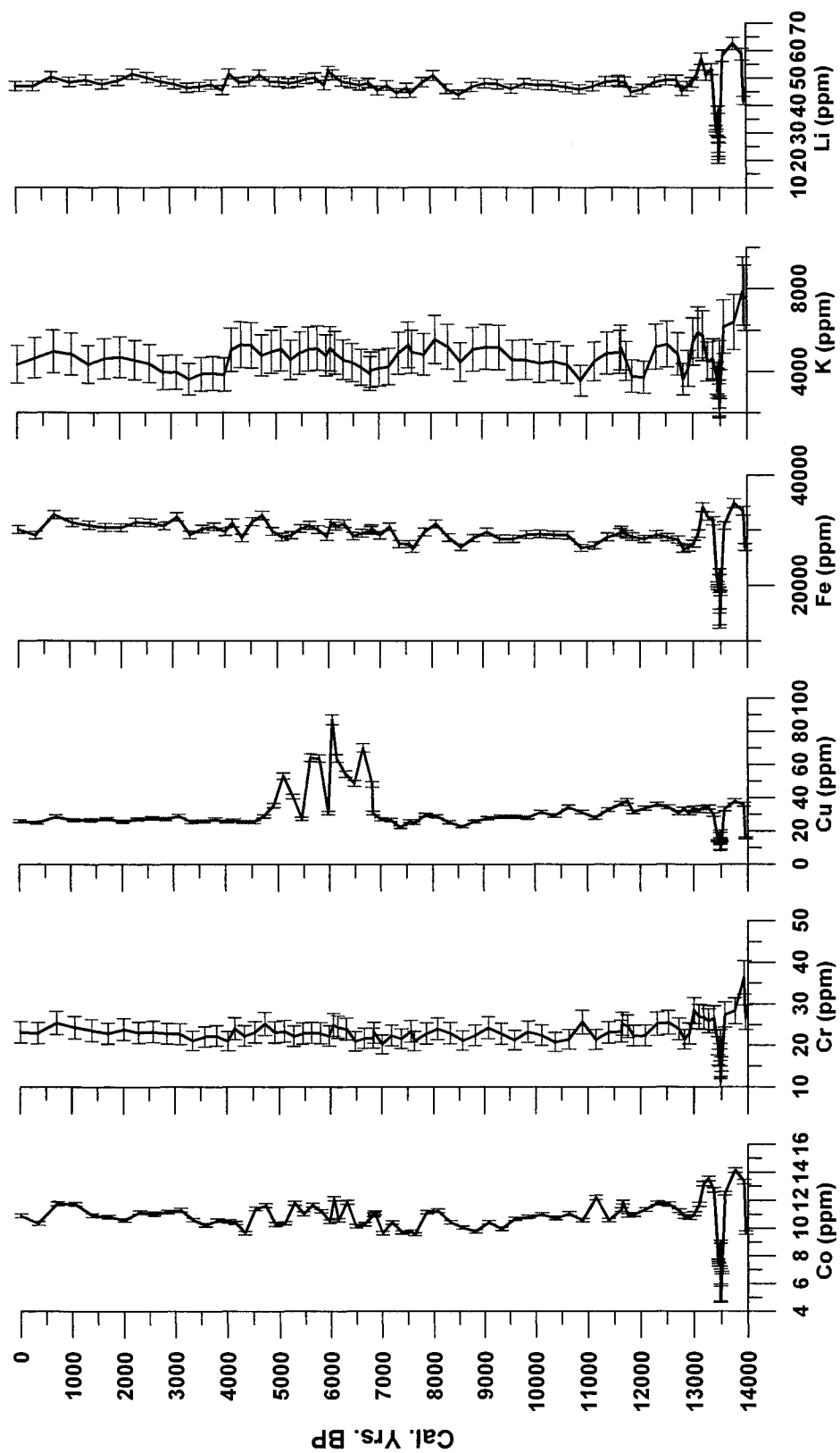


Figure 68 – Core FL-00-9P elemental data (con't).

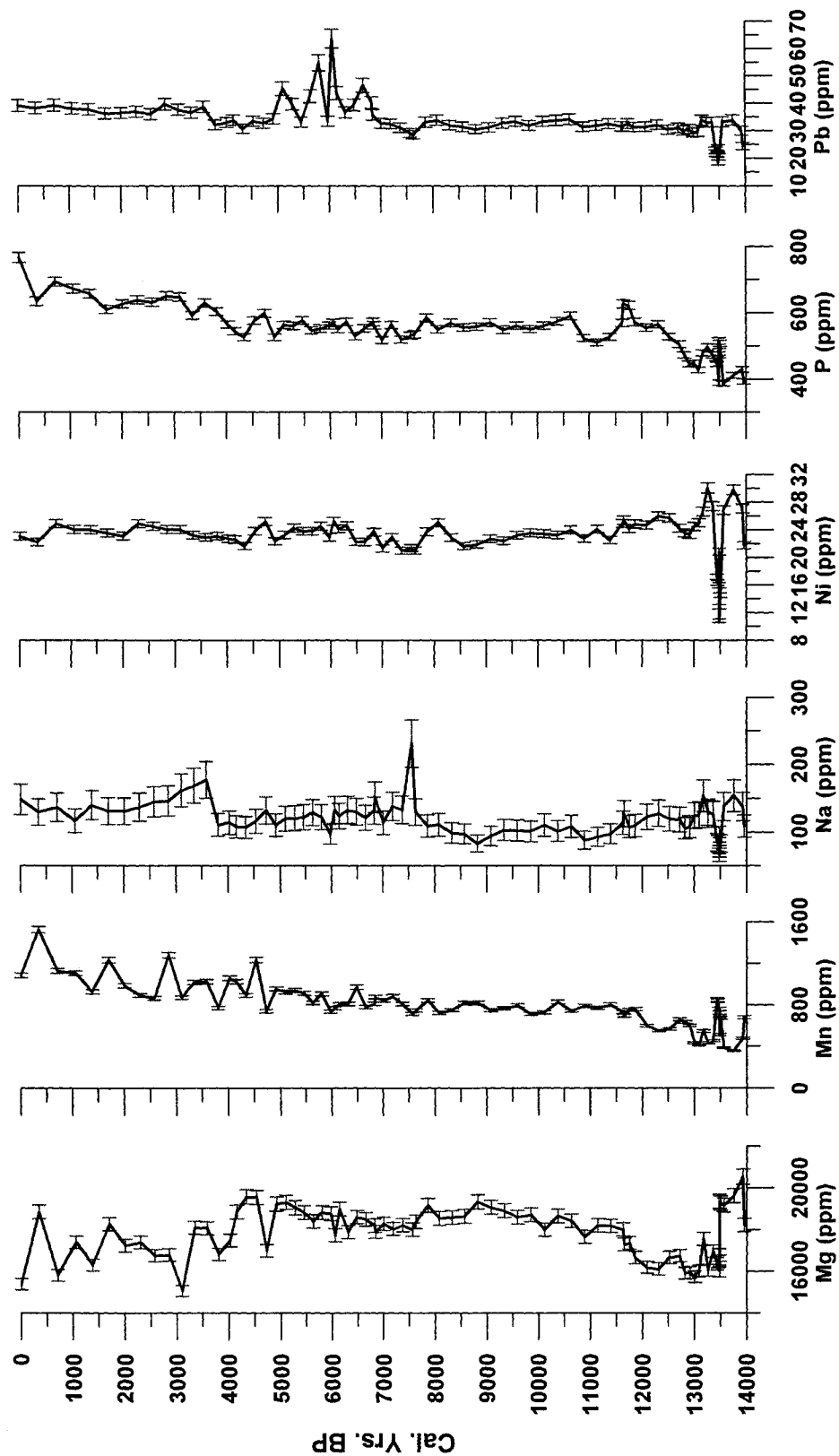


Figure 68 – Core FL-00-9P elemental data (con't).

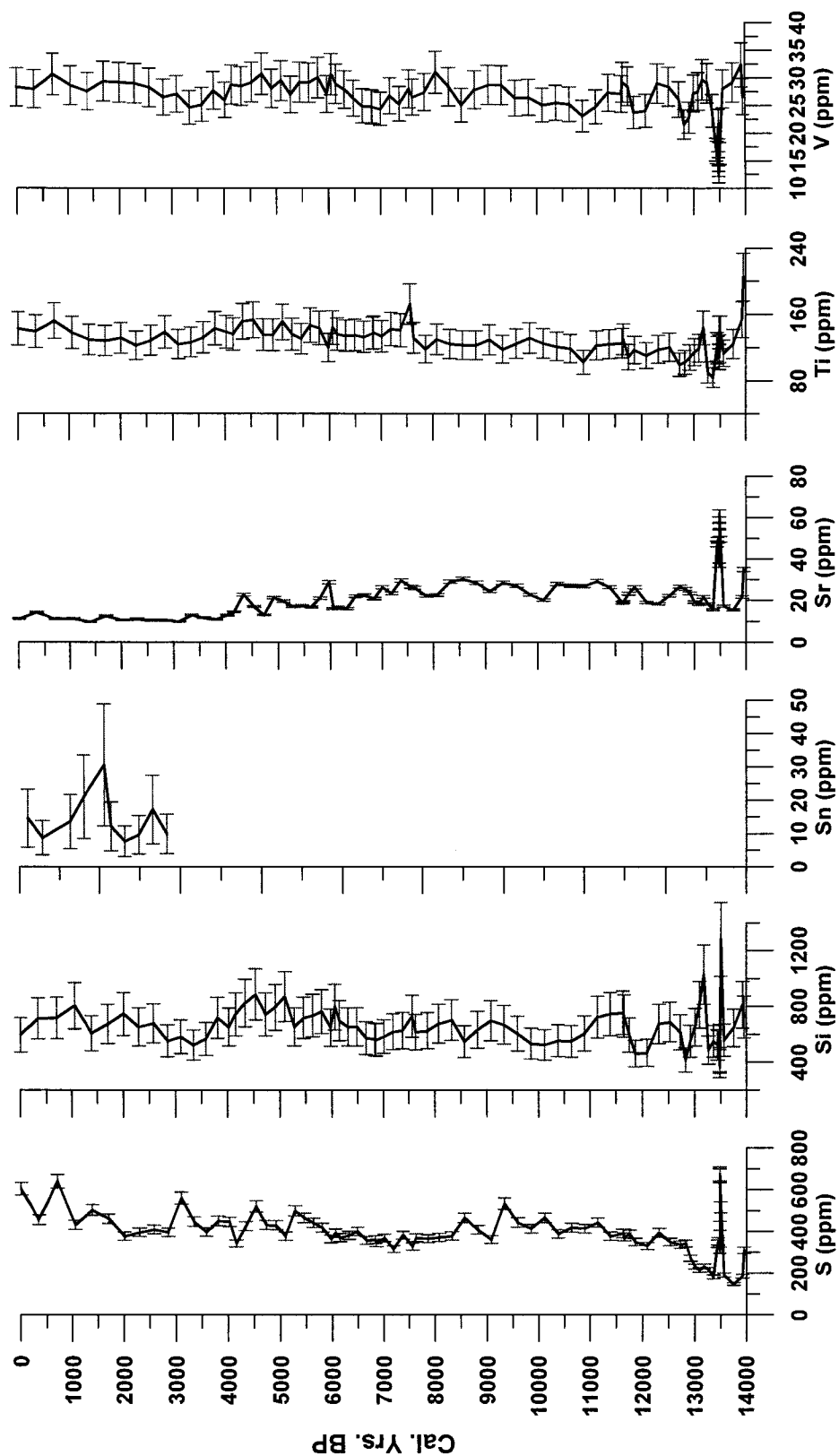


Figure 68 – Core FL-00-9P elemental data (con't).

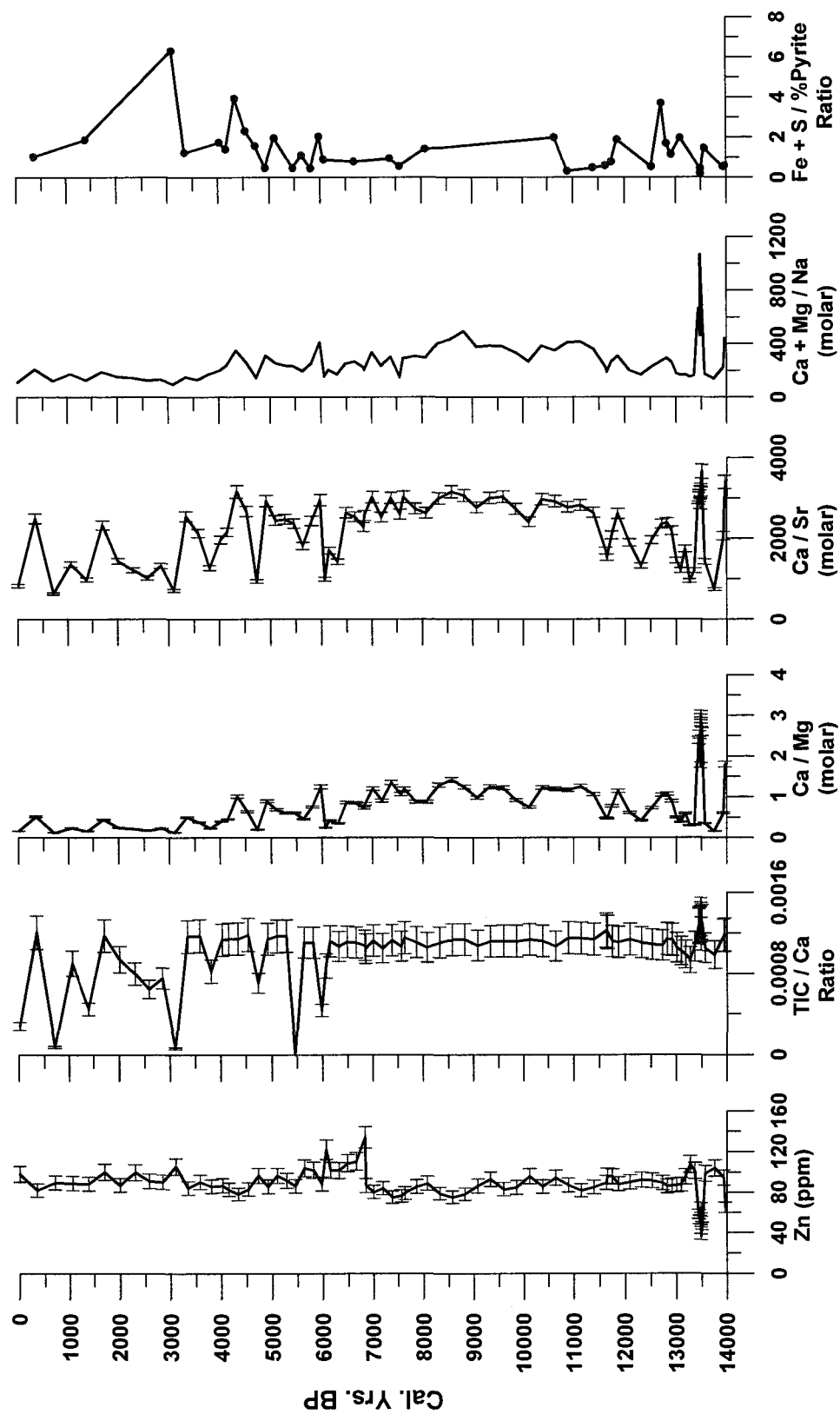


Figure 68 – Core FL-00-9P elemental data (con't).

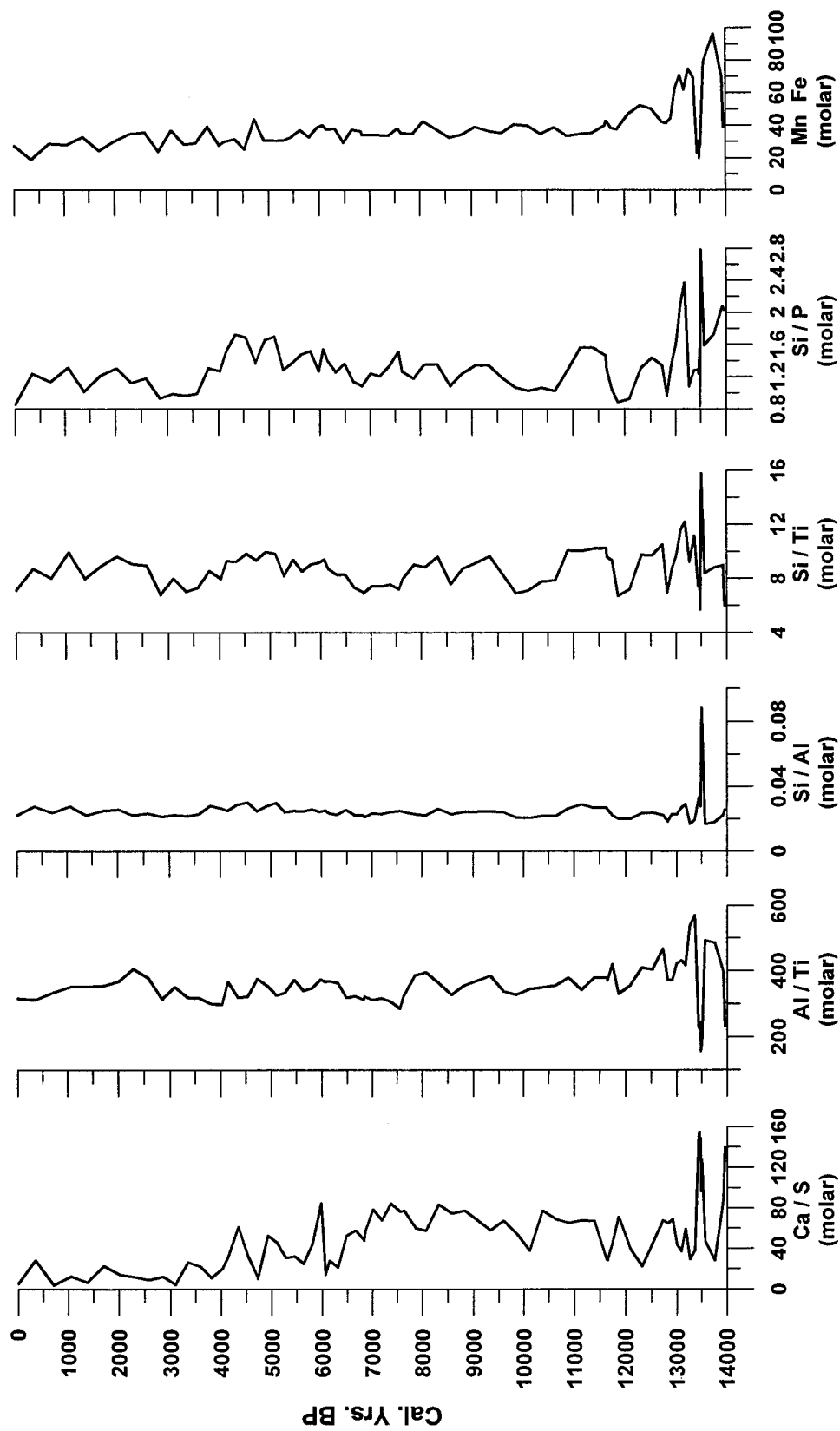


Figure 68 – Core FL-00-9P elemental data (con't).

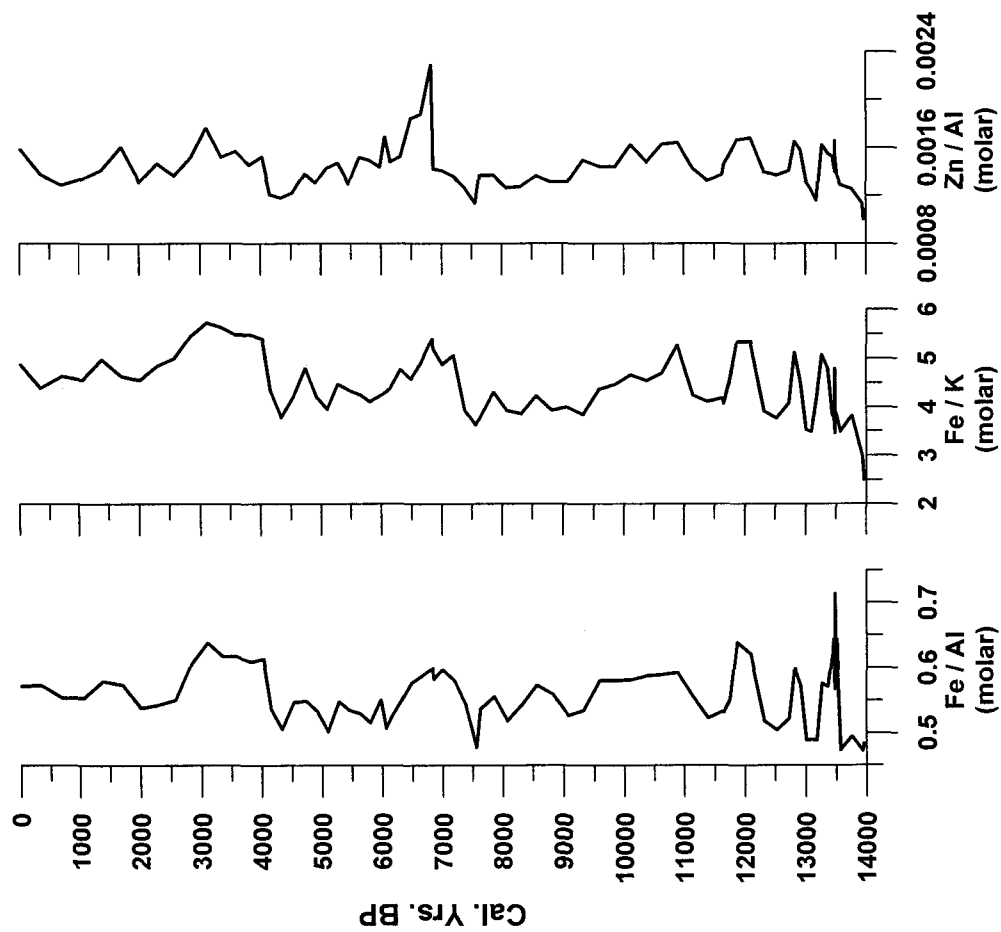


Figure 68 – Core FL-00-9P elemental data (con't).

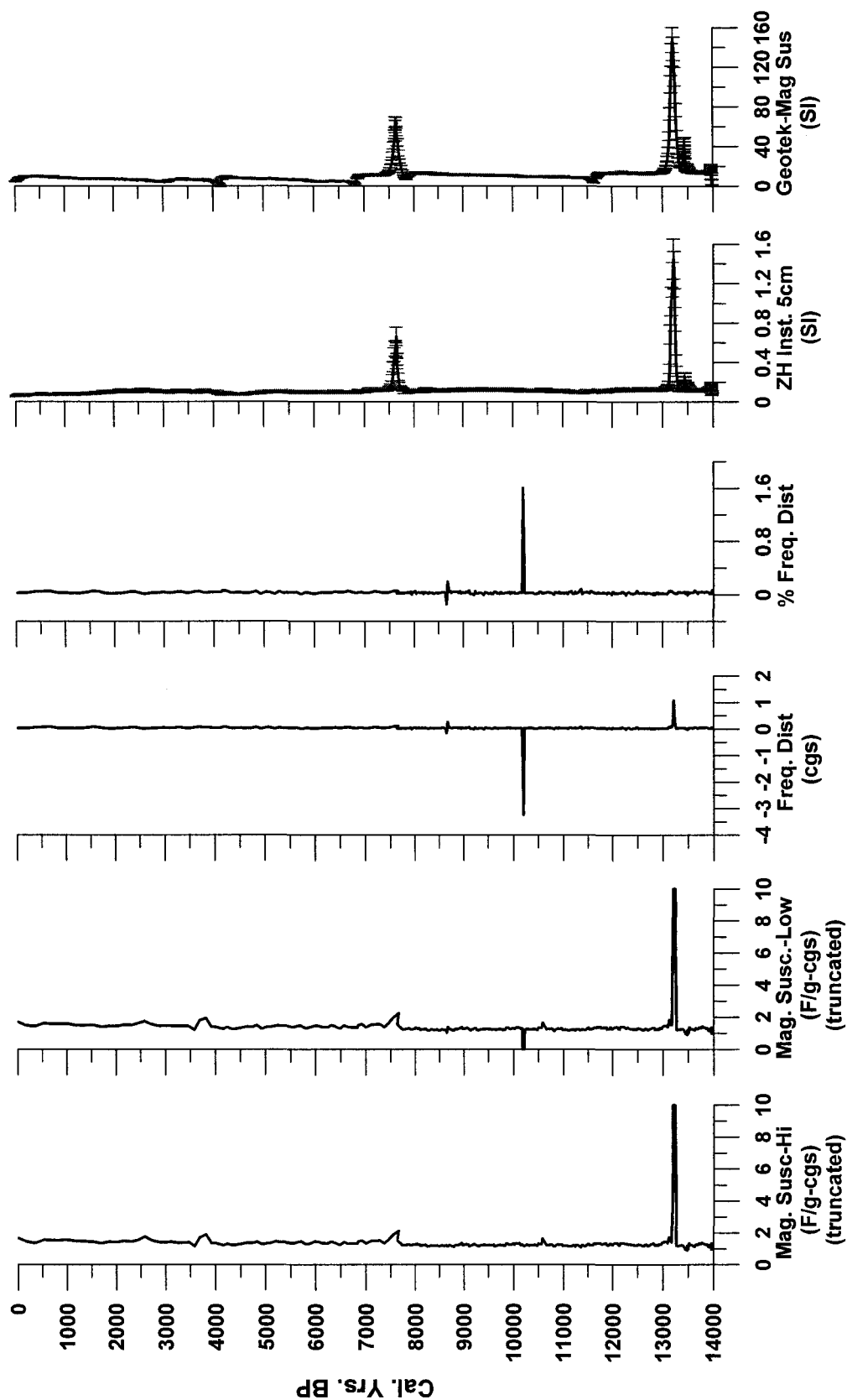


Figure 69 – Magnetic susceptibility measurements for core FL-00-9P. First 4 graphs from Bartington discrete samples, than 5cm integrated measurement on ZH Instruments, last 8cm integrated measurement on Bartington loop reader.

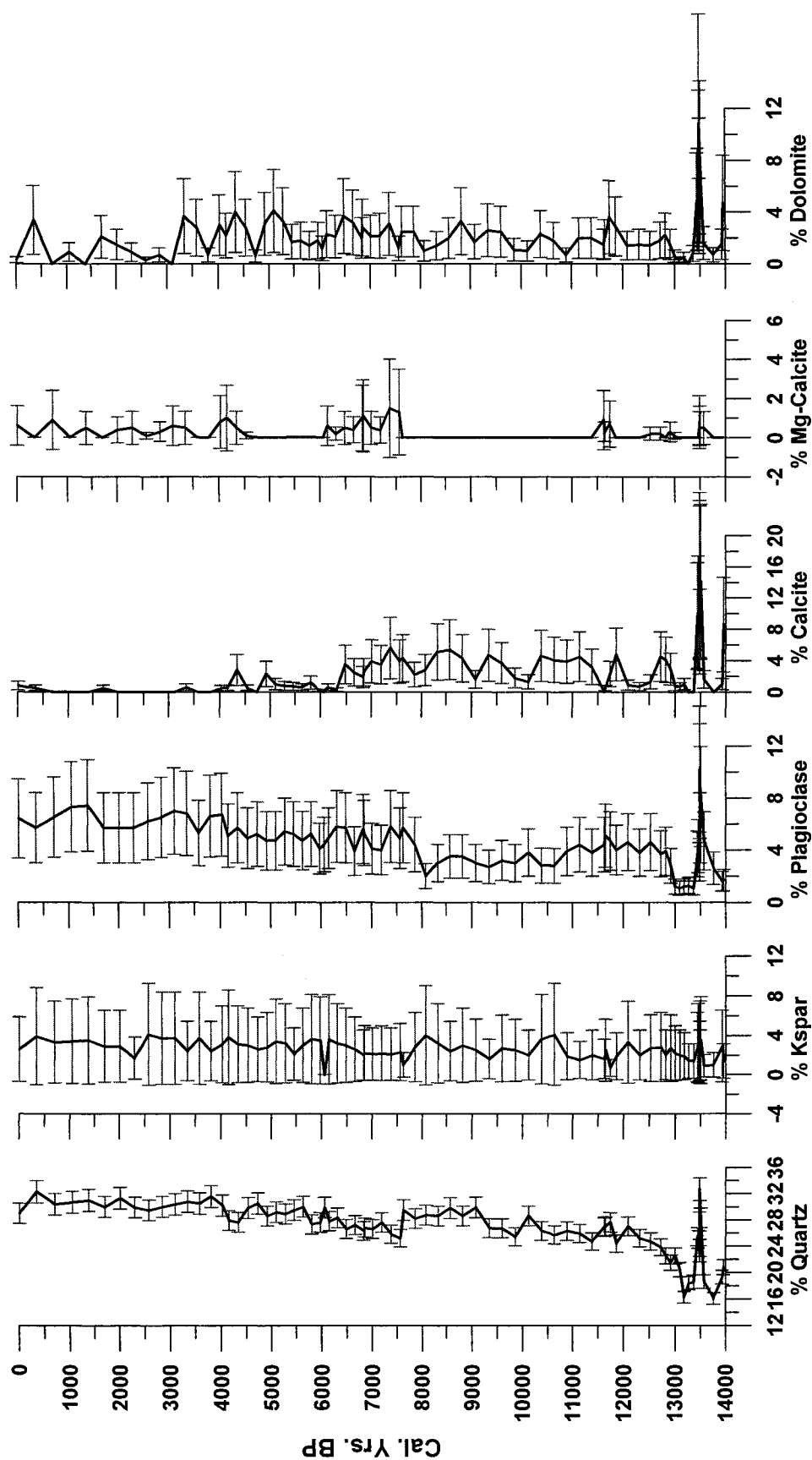


Figure 70 – Core FL-00-9P Mineralogical data.

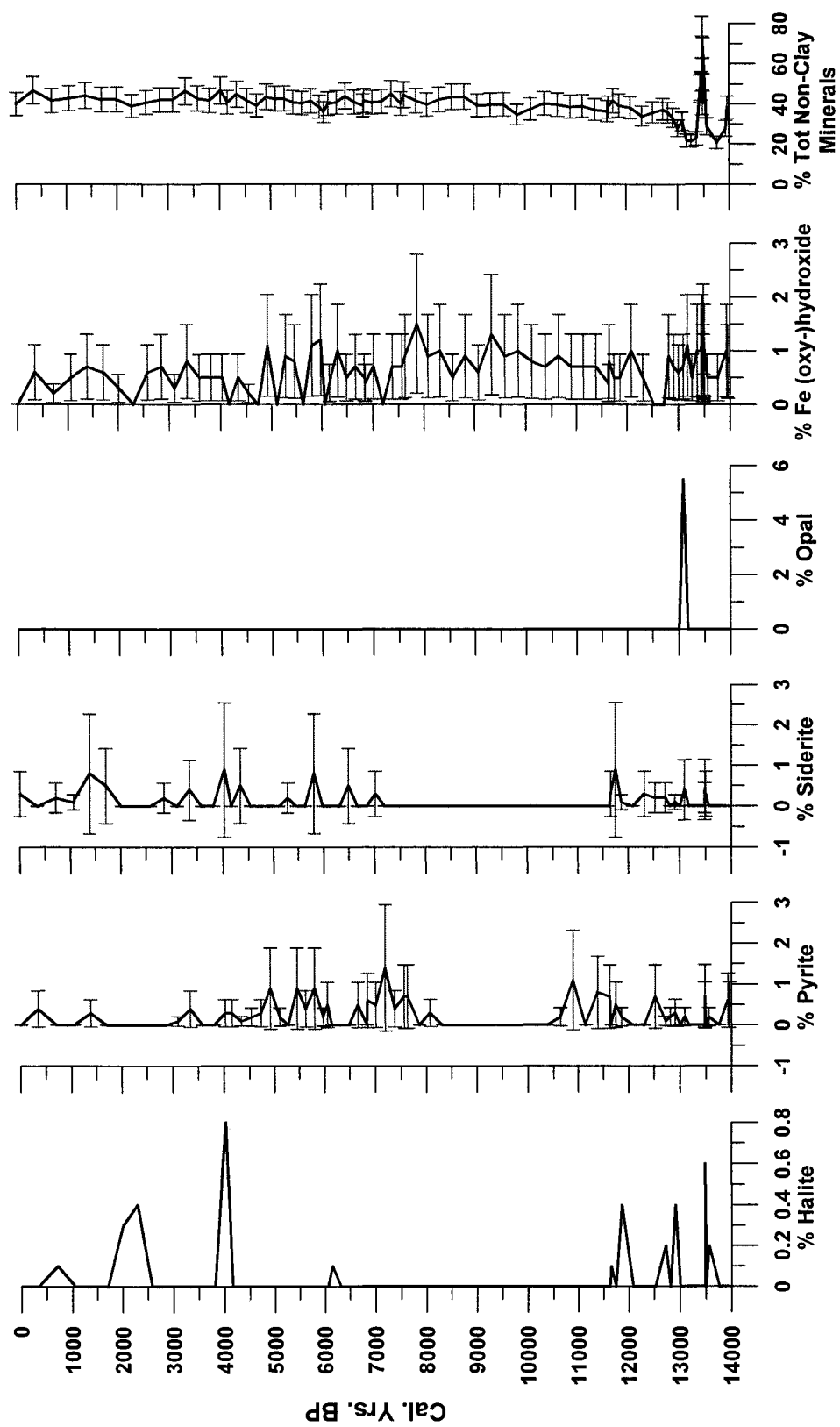


Figure 70 – Core FL-00-9P Mineralogical data (con't).

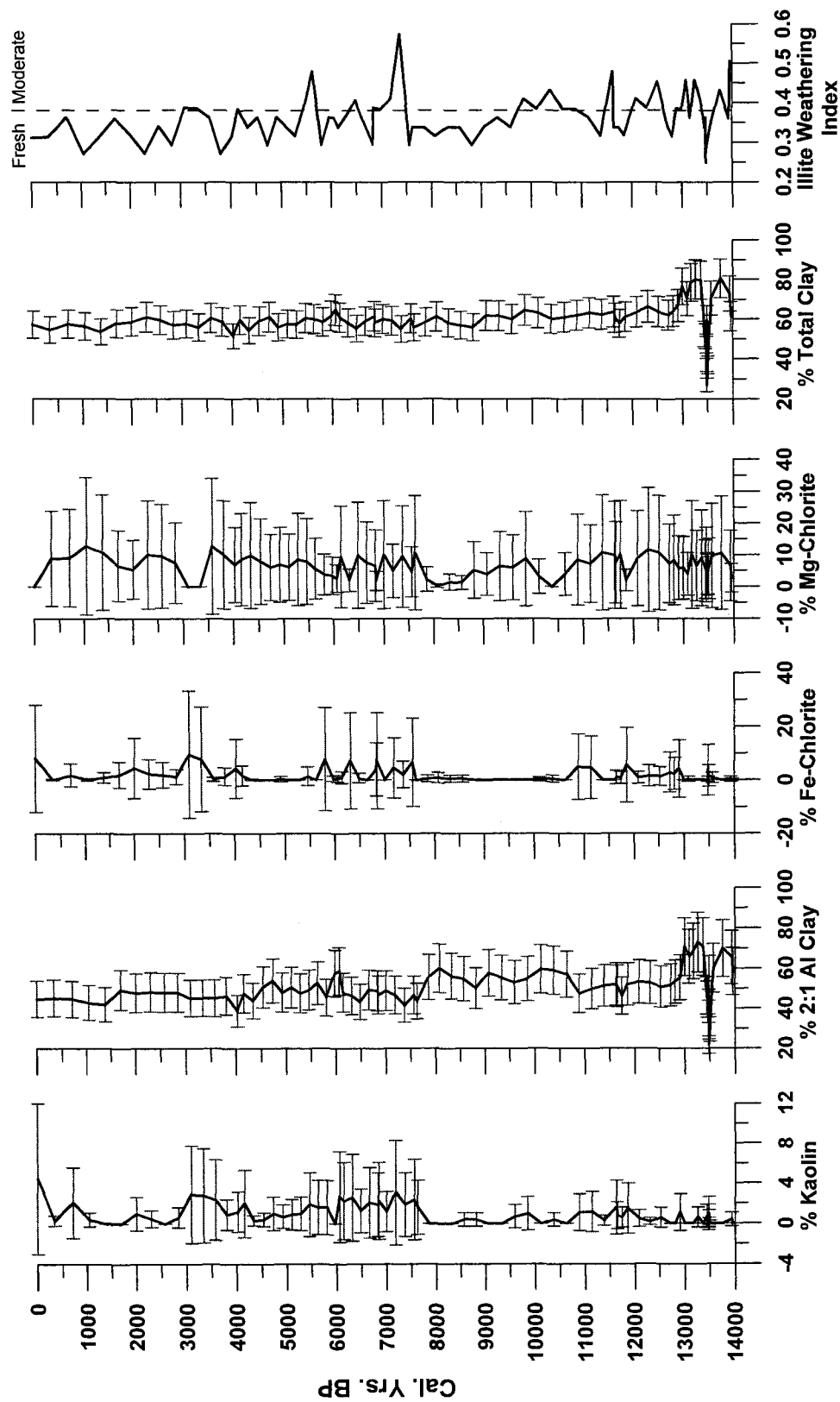


Figure 70 – Core FL-00-9P Mineralogical data (con't).

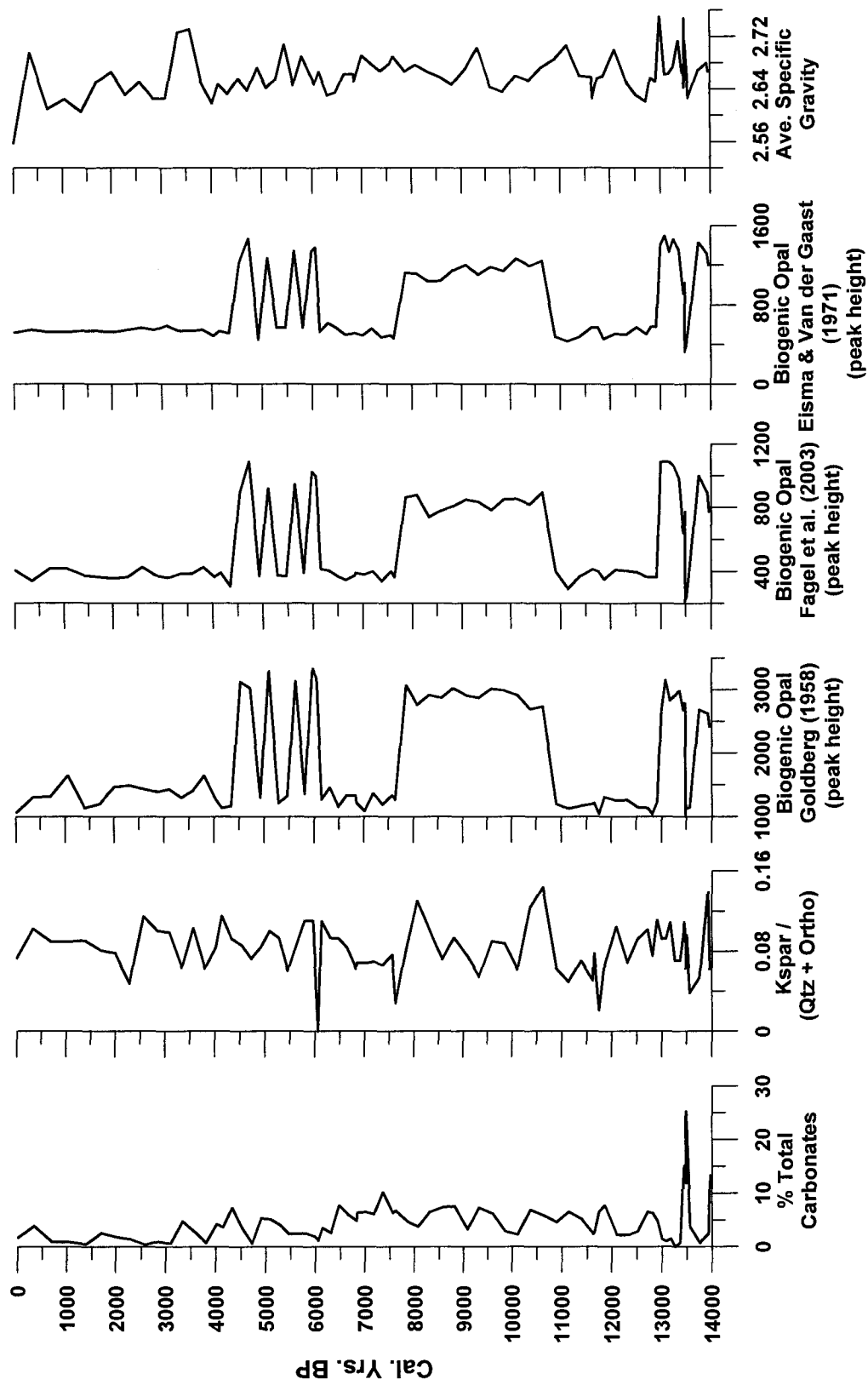


Figure 70 – Core FL-00-9P Mineralogical data (con't).

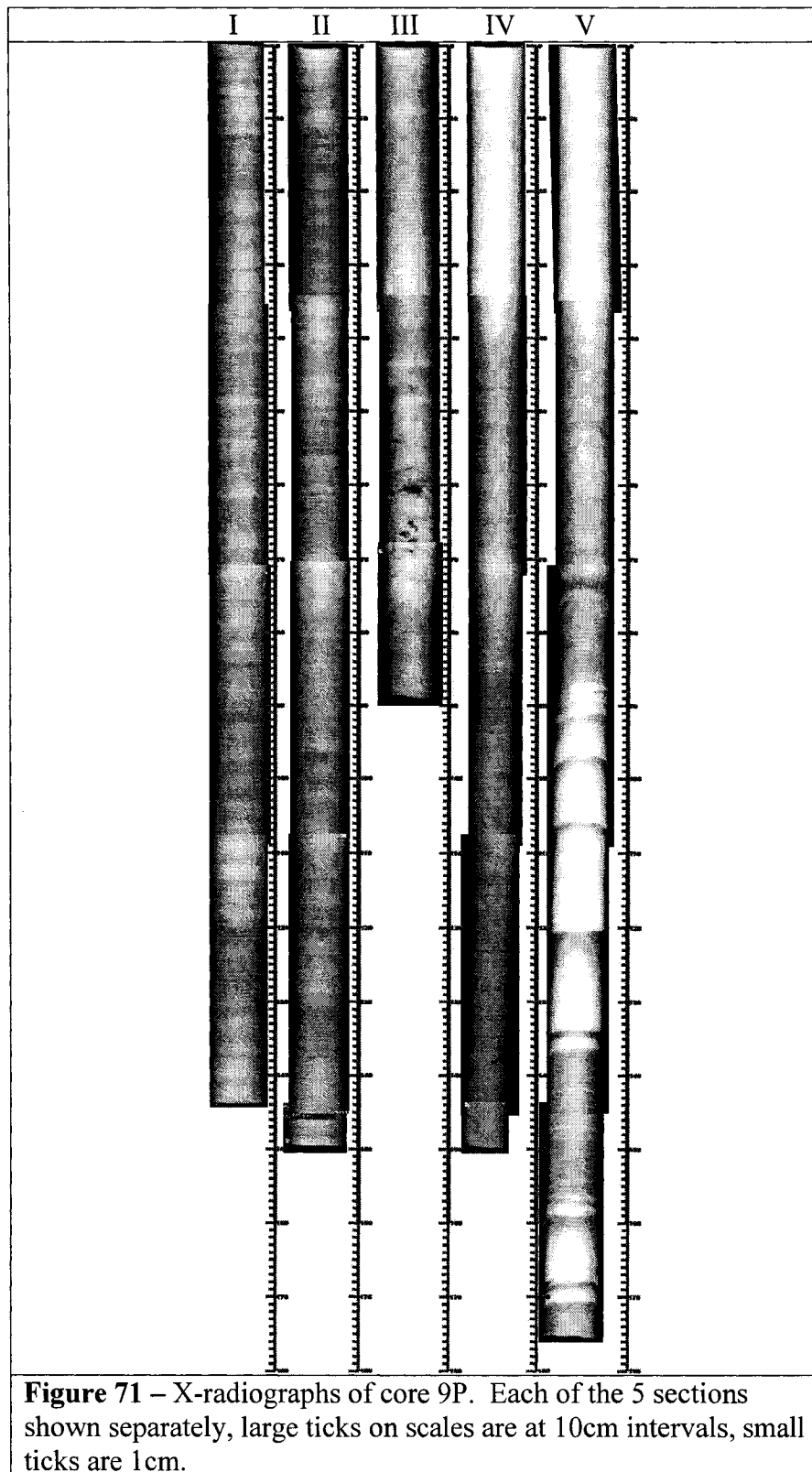


Figure 71 – X-radiographs of core 9P. Each of the 5 sections shown separately, large ticks on scales are at 10cm intervals, small ticks are 1cm.

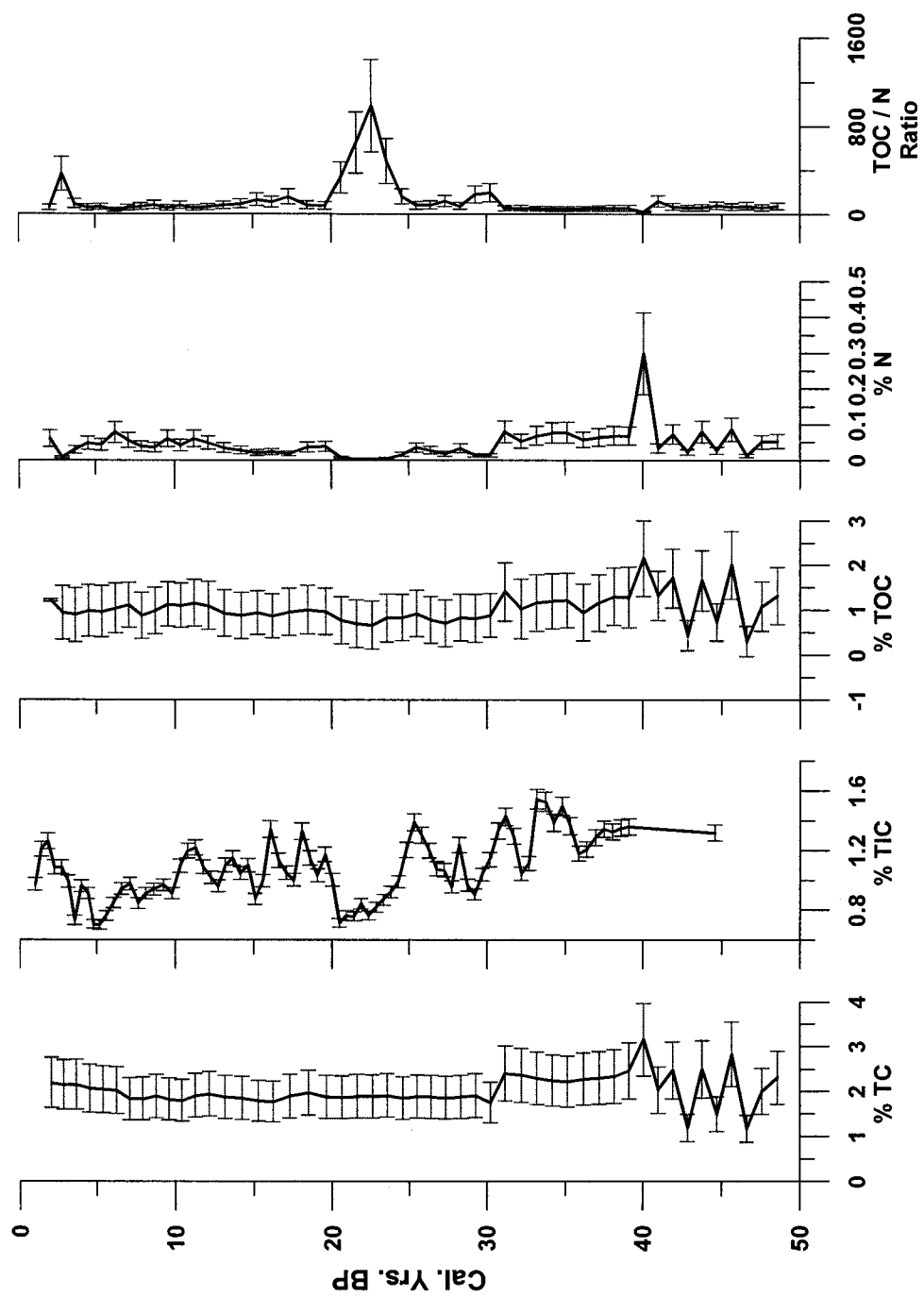


Figure 72 – Core FL-00-9Gb carbon and nitrogen data.

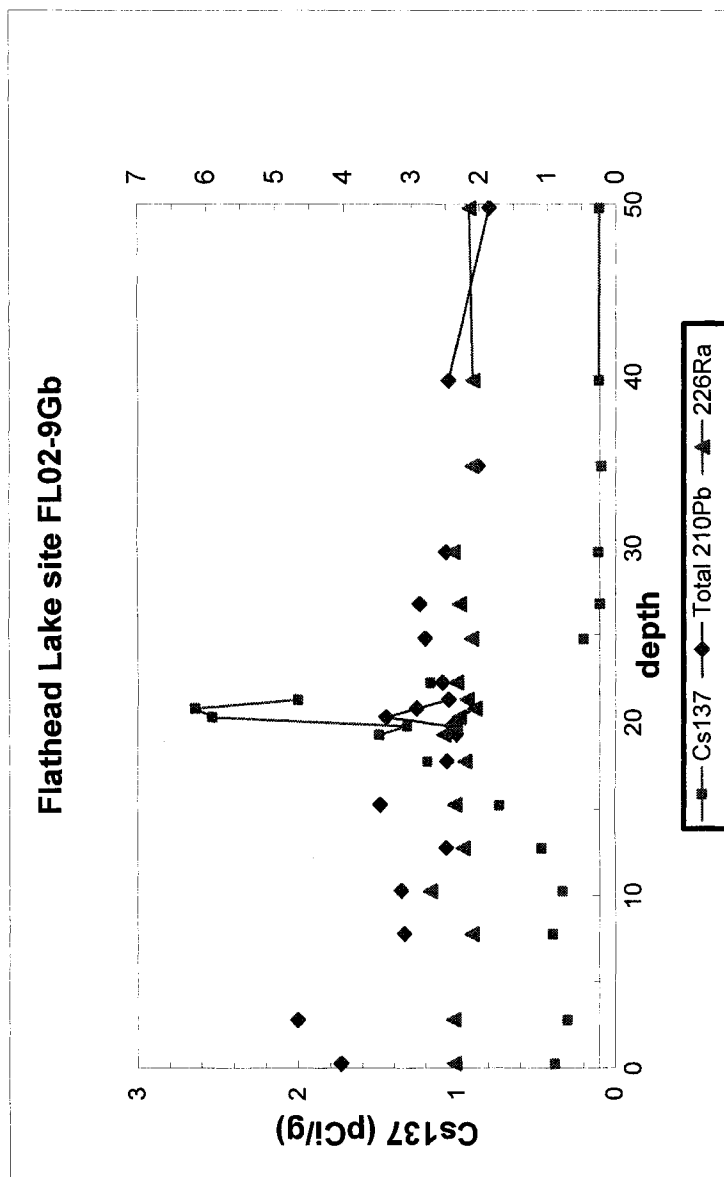


Figure 73 – Core FL-00-9Gb Radiometric Dating Data.

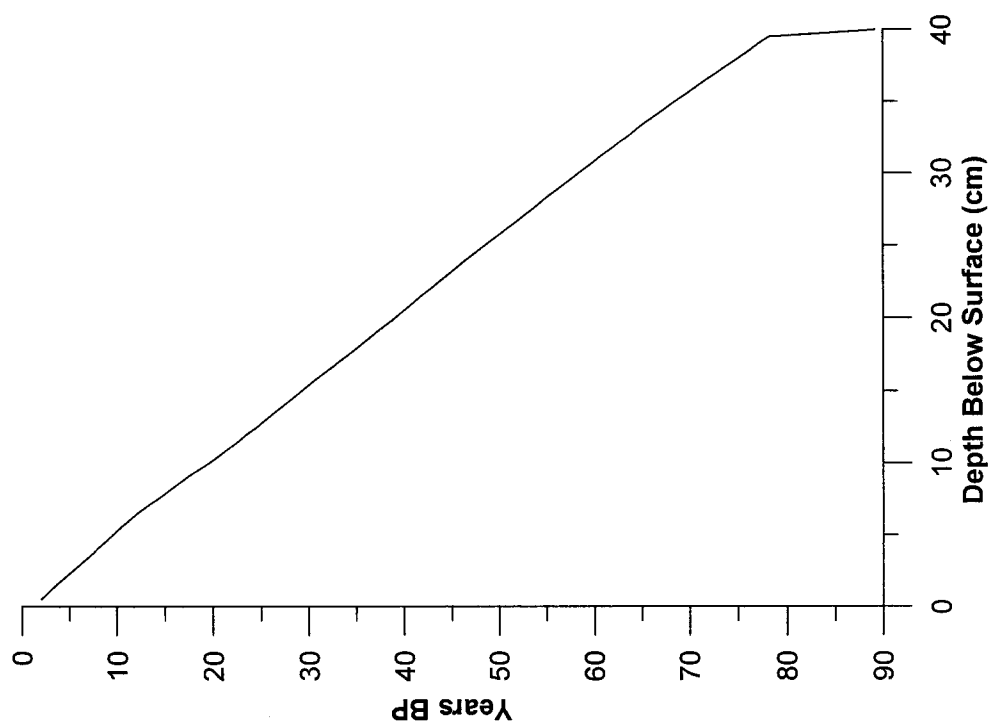


Figure 73 (con't) – Core FL-00-9Gb Dating Model.

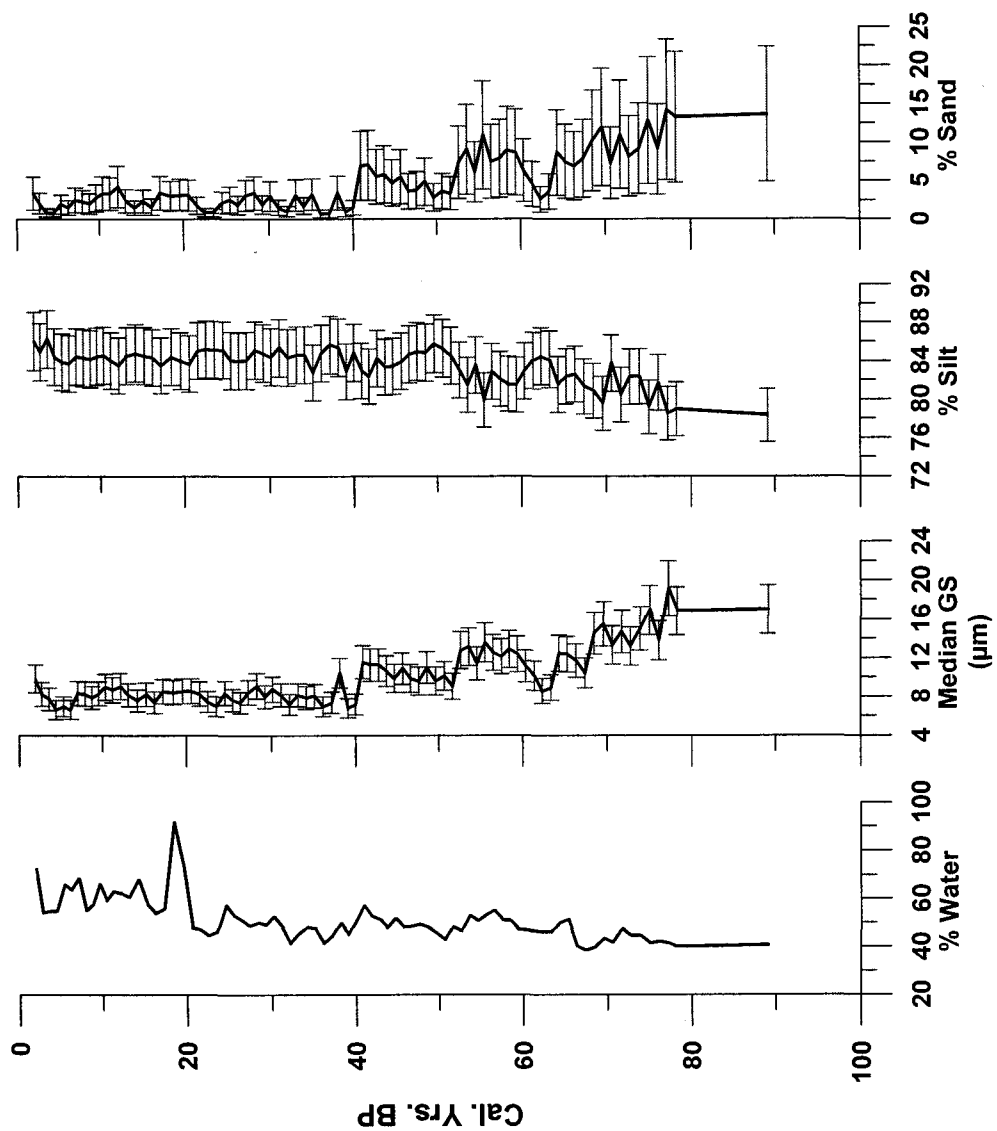


Figure 74 – Core FL-00-9Gb Water and Grain Size Data.

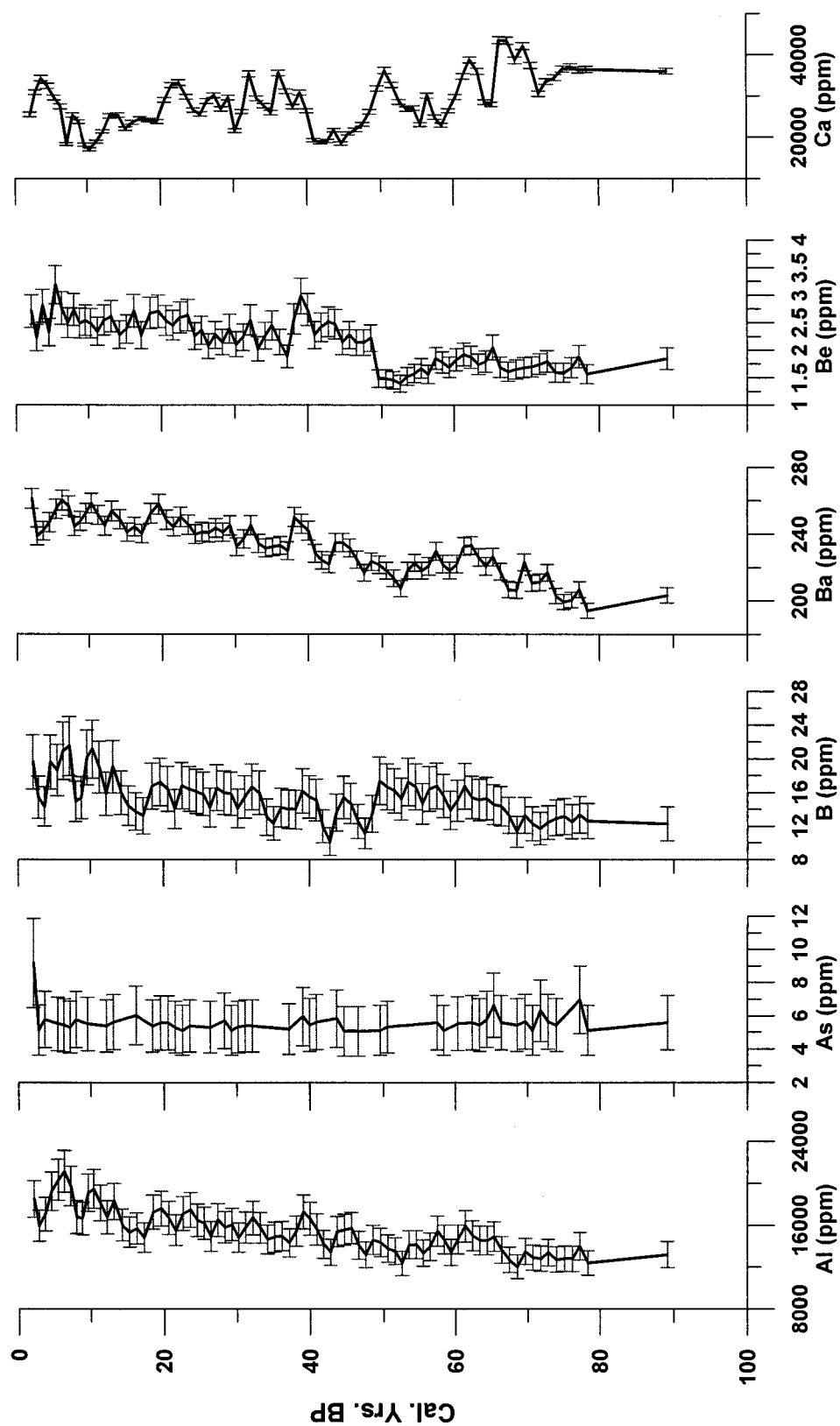


Figure 75 – Core FL-00-9Gb Elemental Data.

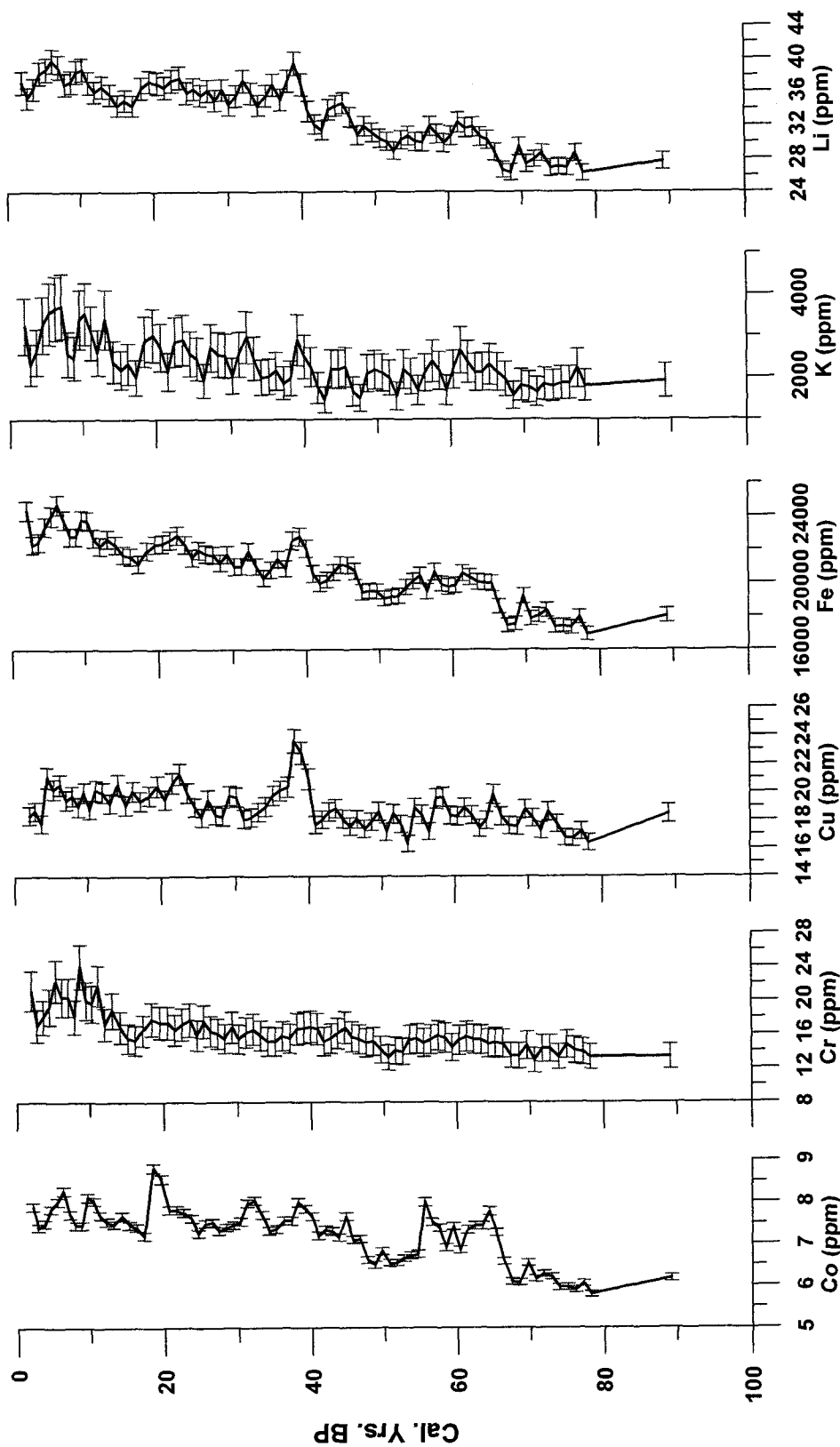


Figure 75 – Core FL-00-9Gb Elemental Data (con't).

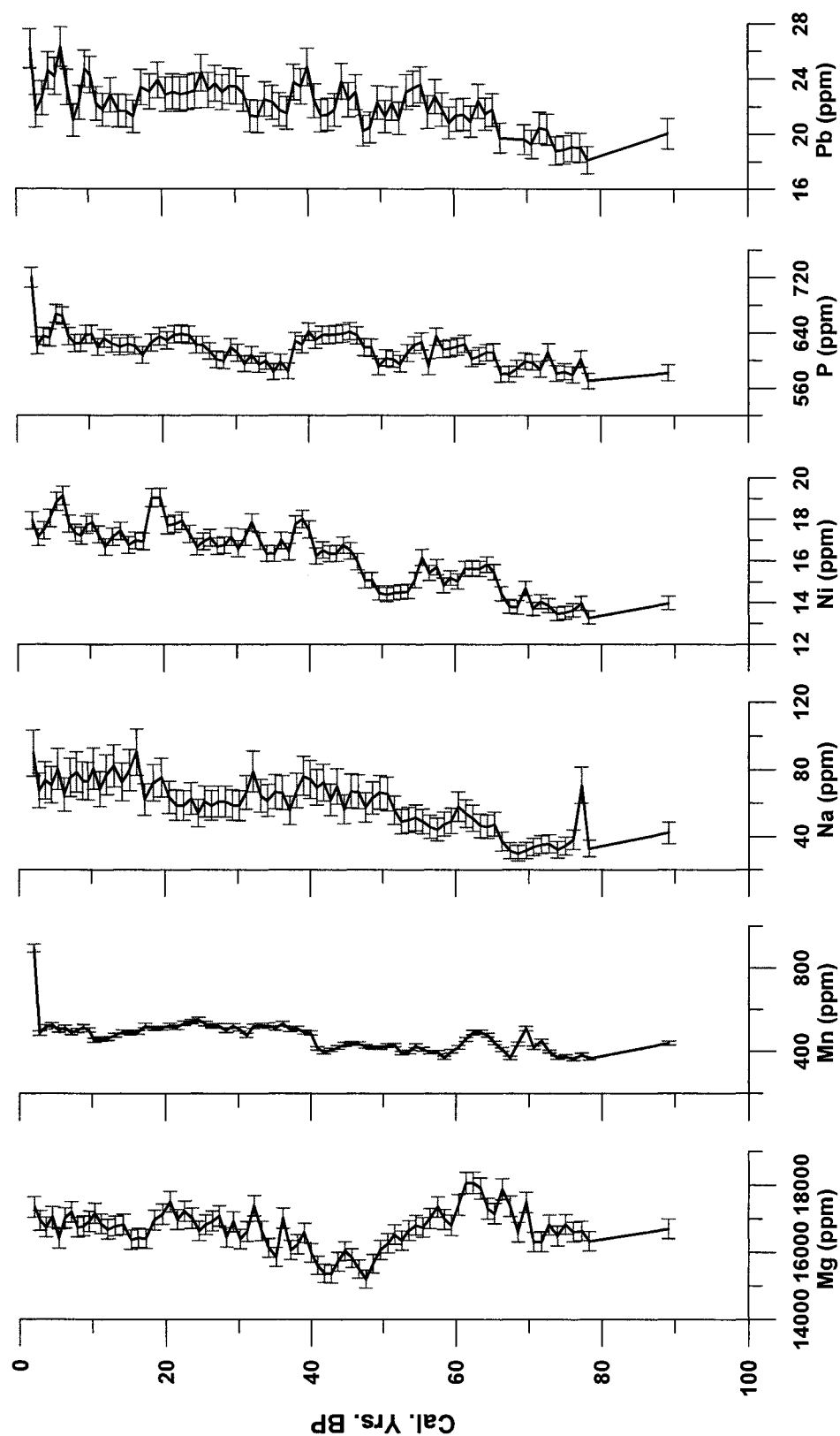


Figure 75 – Core FL-00-9Gb Elemental Data (con't).

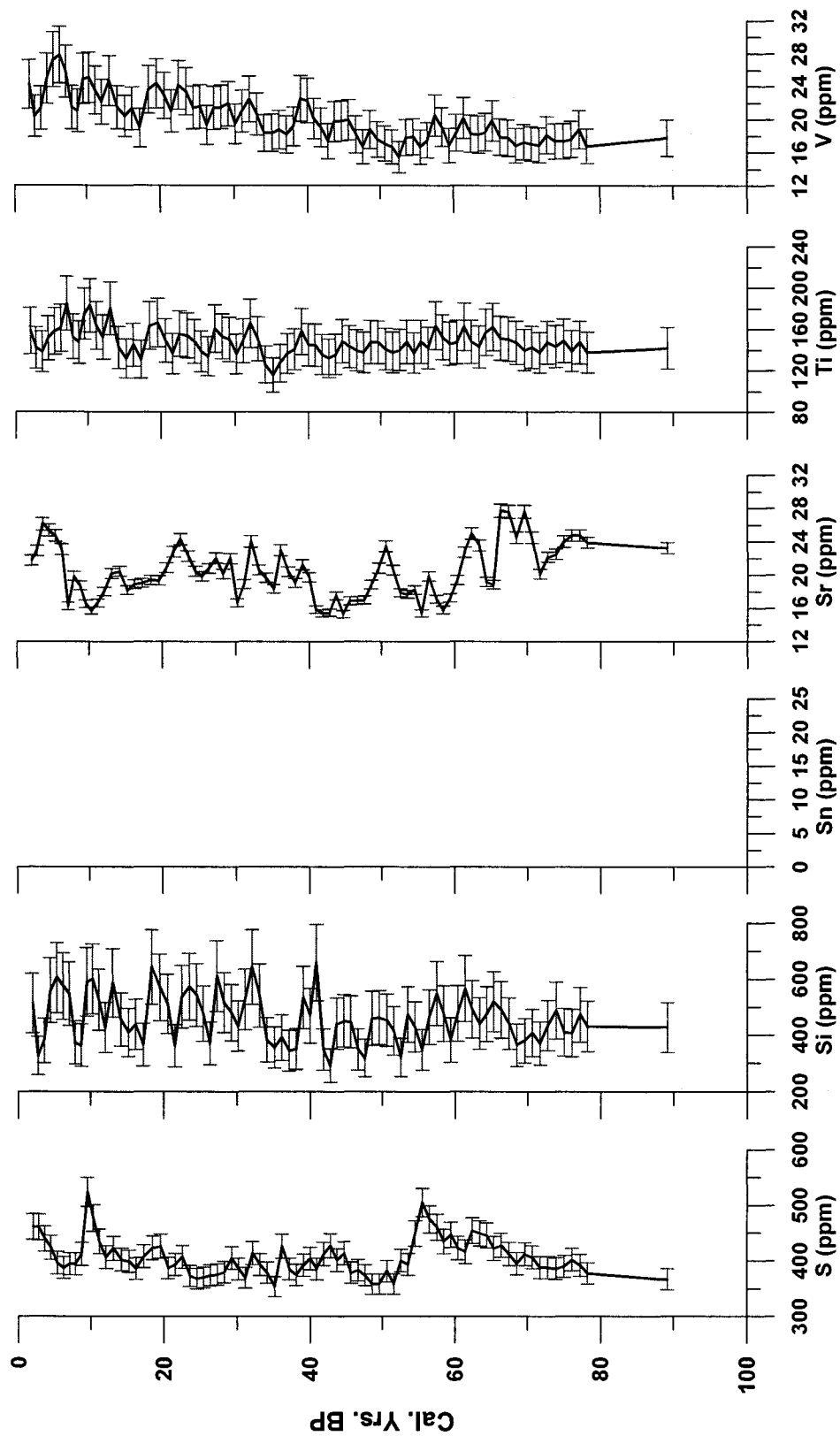


Figure 75 – Core FL-00-9Gb Elemental Data (con't).

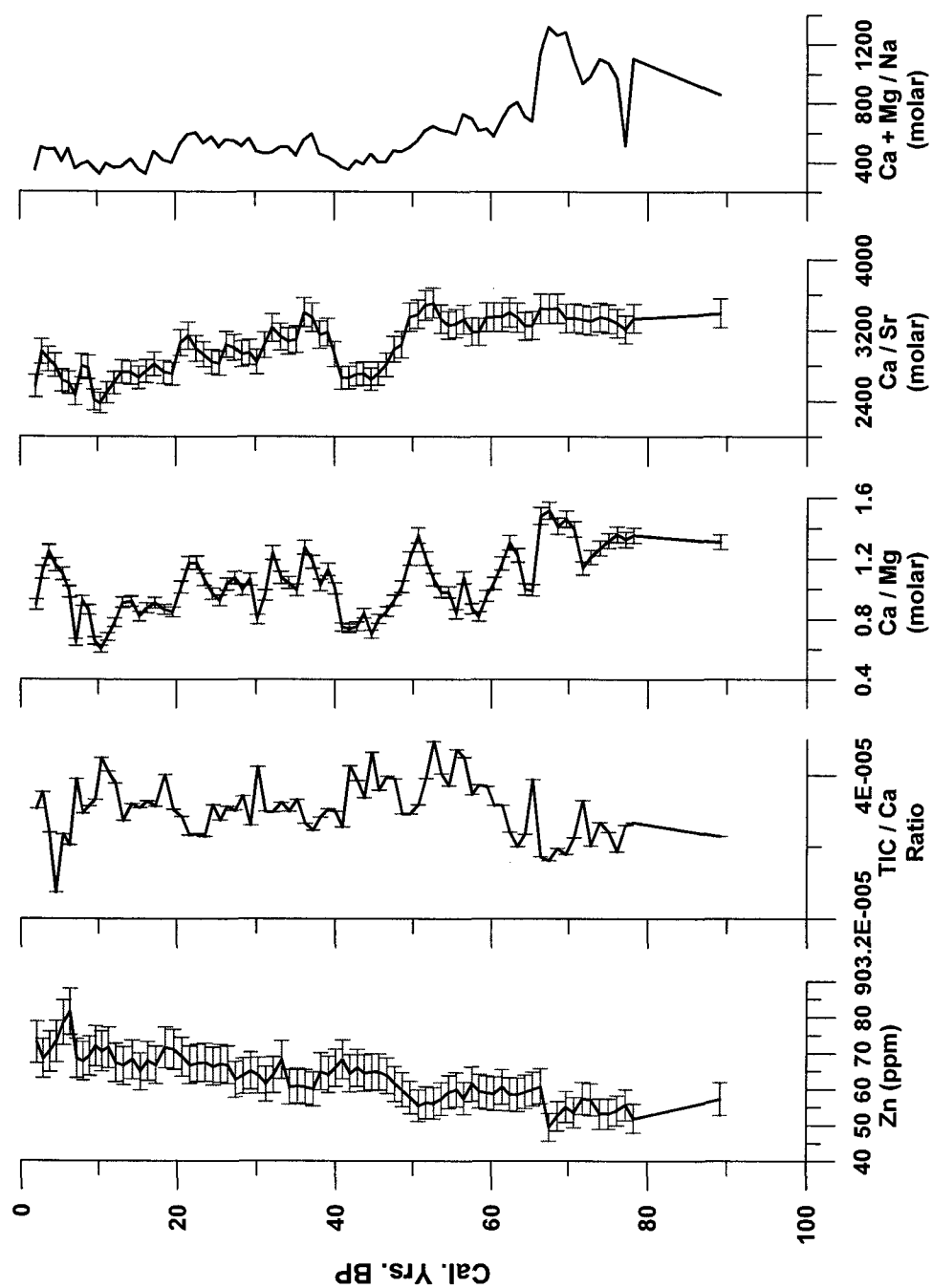


Figure 75 – Core FL-00-9Gb Elemental Data (con't).

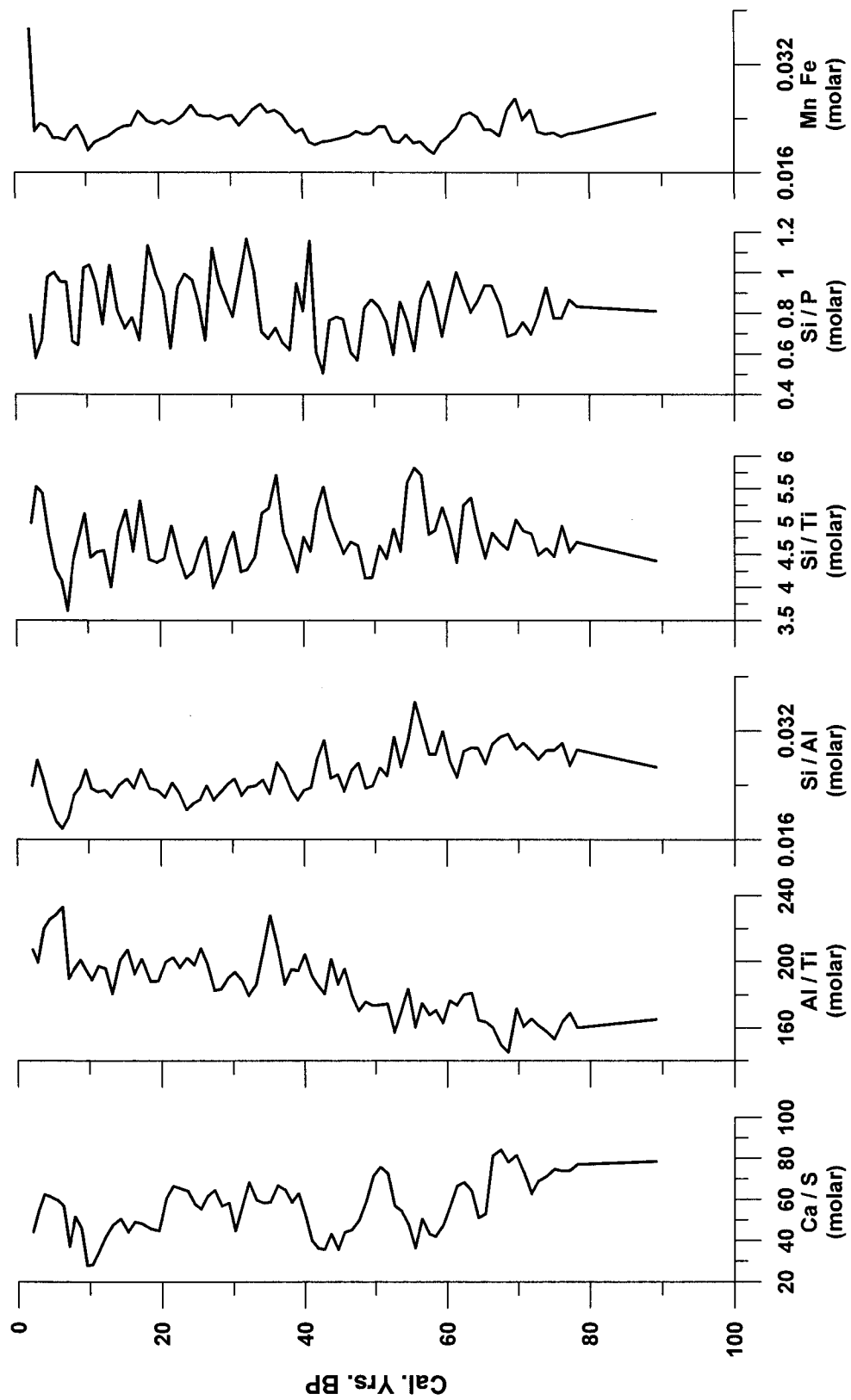


Figure 75 – Core FL-00-9Gb Elemental Data (con't).

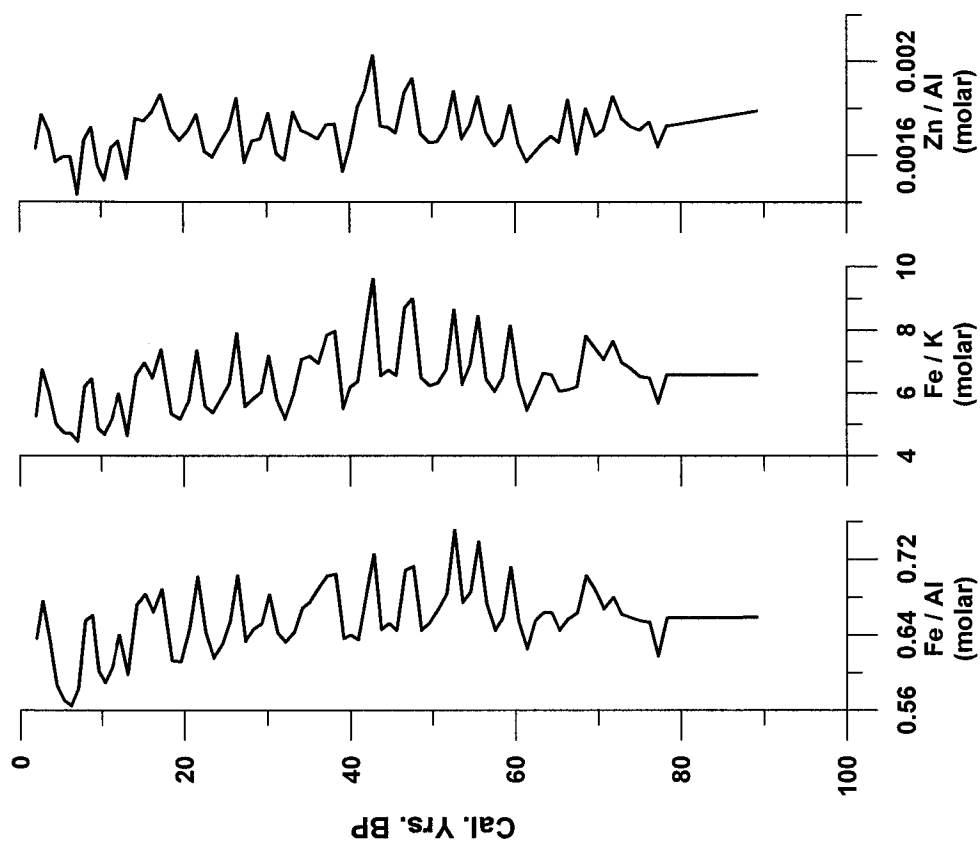


Figure 75 – Core FL-00-9Gb Elemental Data (con't).

DESCRIPTION OF PLATES

Photographic plates provide a representative sampling of SEM images taken during qualitative analysis of sediments. A full set of the SEM images and EDS analyses can be found in Appendix F. Dr. Jeffery Stone, University of Nebraska-Lincoln performed all identifications of diatoms species or genera found on plates 1 and 2.

Plate 1:

- a) FL-03-16K-II-129 Photo 12, image of benthic, shallow water diatom Naviculoid, possibly *Anomoeoneis costata*.
- b) FL-03-16K-II-129 Photo 28, image of benthic, shallow water diatom Naviculoid, possibly *Anomoeoneis costata*.
- c) FL-03-16K-II-129 Photo 17, image of benthic, shallow water diatom *Campylodiscus*, probably *Campylodiscus hibernicus*.
- d) FL-03-16K-II-129 Photo 17-2, close up of *Campylodiscus*, probably *Campylodiscus hibernicus* shown in image c.
- e) FL-03-16K-II-129 Photo 16, image of benthic, shallow water diatom *Cymatopleura solea* v. *apiculata*.
- f) FL-03-16K-II-129 Photo 6, image of benthic, shallow water diatom *Staurosira construens* v. *venter*.

Plate 2:

- a) FL-03-16K-II-129 Photo 15, image of benthic, shallow water diatom
Pseudostaurosira pseudoconstruens.
- b) FL-03-16K-II-129 Photo 24, image of tychoplantic diatom *Aulacoseira*, probably
Aulacoseira distans.
- c) FL-03-16K-II-129 Photo 22, image of tychoplantic diatom *Aulacoseira*, possibly
Aulacoseira italica or *Aulacoseira distans*.
- d) FL-03-16K-II-129 Photo 7, image of planktic, deeper water diatom *Cyclotella*,
probably *Cyclotella ocellata*.
- e) FL-03-16K-II-129 Photo 2, image of planktic, deeper water diatom *Cyclotella*/ sp.
- f) FL-03-16K-II-129 Photo 18, image of planktic, deeper water diatom *Cyclotella*
ocellata.

Plate 3:

- a) FL-00-9P-I-2 Photo 6, image of detrital quartz with some clay plates. EDS
analysis of center of view shown in Figure 76.
- b) FL-00-9P-III-30 Photo 1, image cluster of detrital illite plates. EDS analysis of
cluster shown in Figure 77.
- c) FL-00-9P-III-30 Photo 5, field are image of sample showing quartz grain (middle)
surrounded by clays. EDS analysis of field view shown in Figure 78.
- d) FL-00-9P-IV-10 Photo 3, image of detrital illite. EDS analysis of clay in center of
view shown in Figure 79.

- e) FL-00-9P-IV-10 Photo 6, image of detrital quartz with plates of detrital illite. EDS analysis of center of view shown in Figure 80.
- f) FL-00-9P-V-10B Photo 5, image of detrital dolomite. EDS analysis of center of view shown in Figure 81.

Plate 4:

- a) FL-00-9P-V-120 Calcite1, image of detrital calcite grain with clay coating. EDS analysis of grain shown in Figure 82.
- b) FL-00-9P-V-120 Cal4, image of detrital calcite grain with clay coating. EDS analysis of grain shown in Figure 83.
- c) FL-00-9P-V-120 Dolomite, image of detrital dolomite with coating of clay. EDS analysis of grain shown in Figure 84.
- d) FL-03-16K-II-145 Photo 1, image of Mt. Mazama tephra. EDS analysis of ash grain in center of view shown in Figure 85.
- e) FL-03-16K-II-145 Photo 8, image of Mt. Mazama tephra. EDS analysis of ash grain in center of view shown in Figure 86.
- f) FL-03-15K-V-161 Photo 1, image of detrital clays from glacial varve. EDS analysis of field view shown in Figure 87.

Plate 1

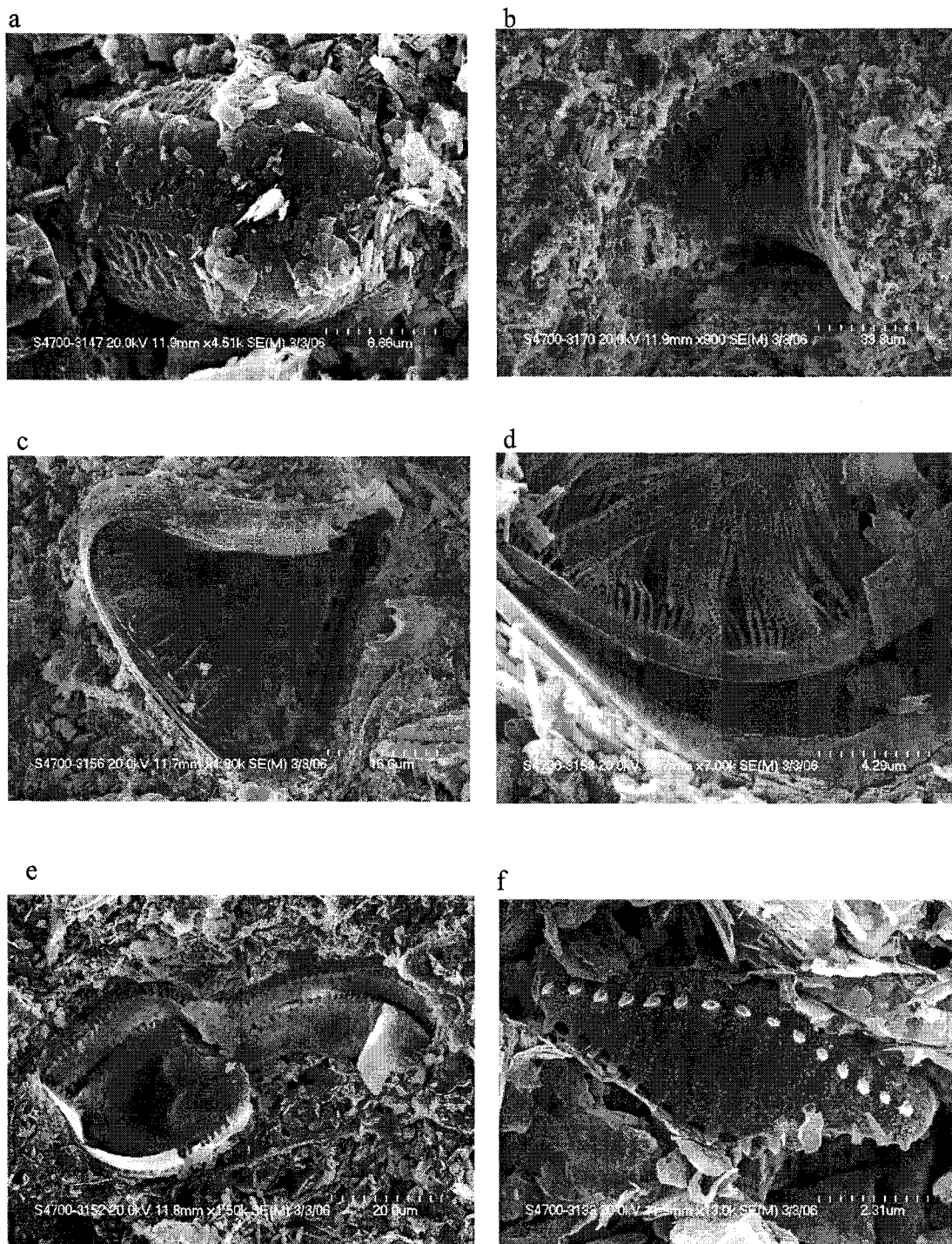


Plate 2

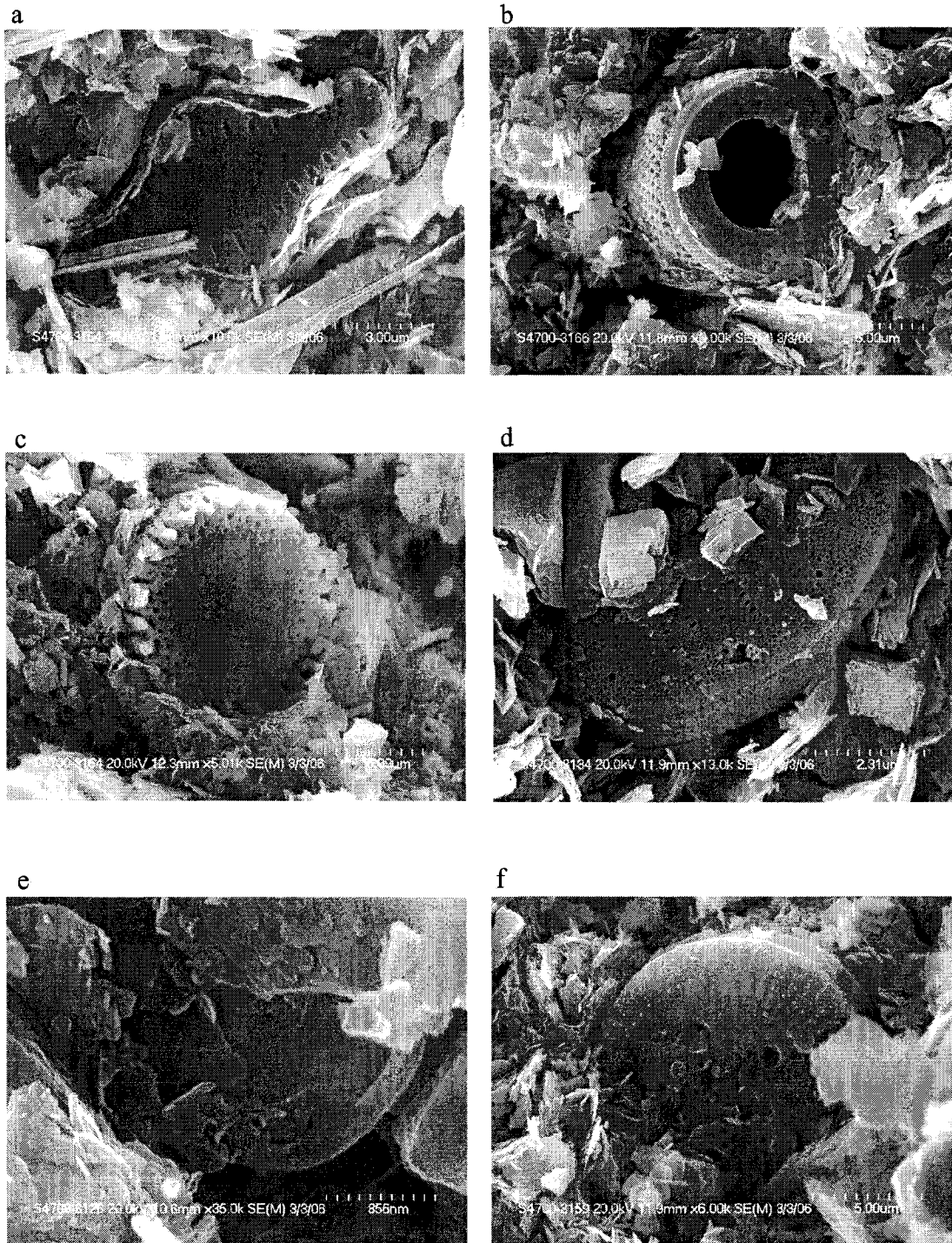


Plate 3

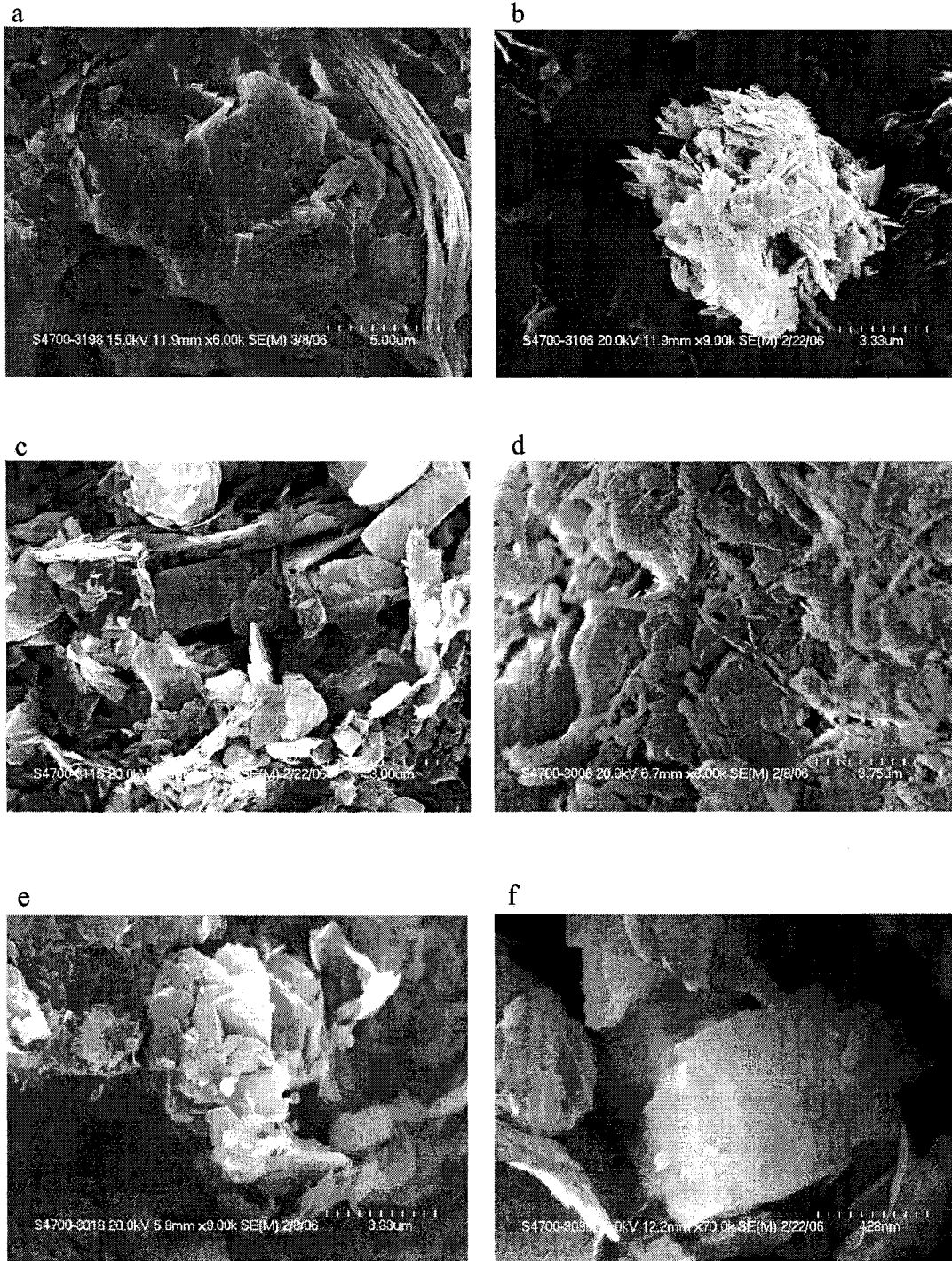
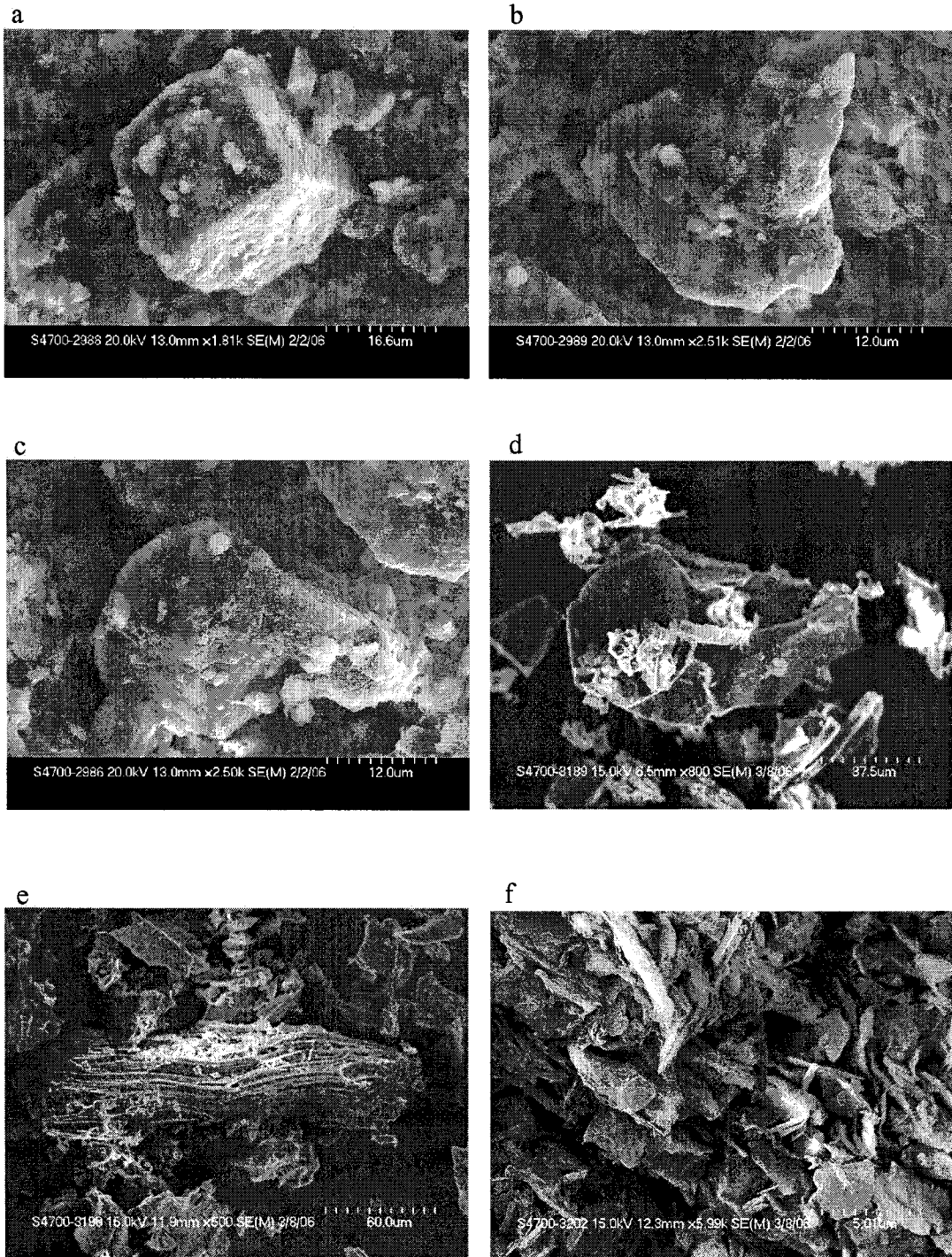


Plate 4



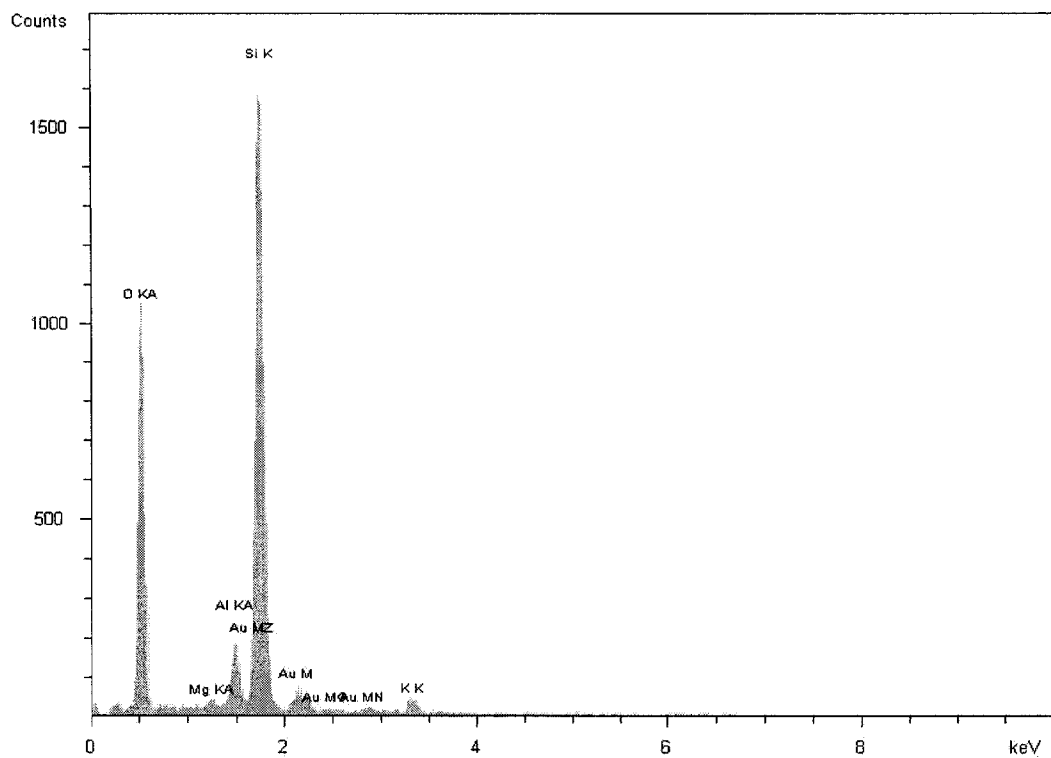


Figure 76 – EDS graph for sample FL-00-9P-II-2 Photo 6, plate 3a.

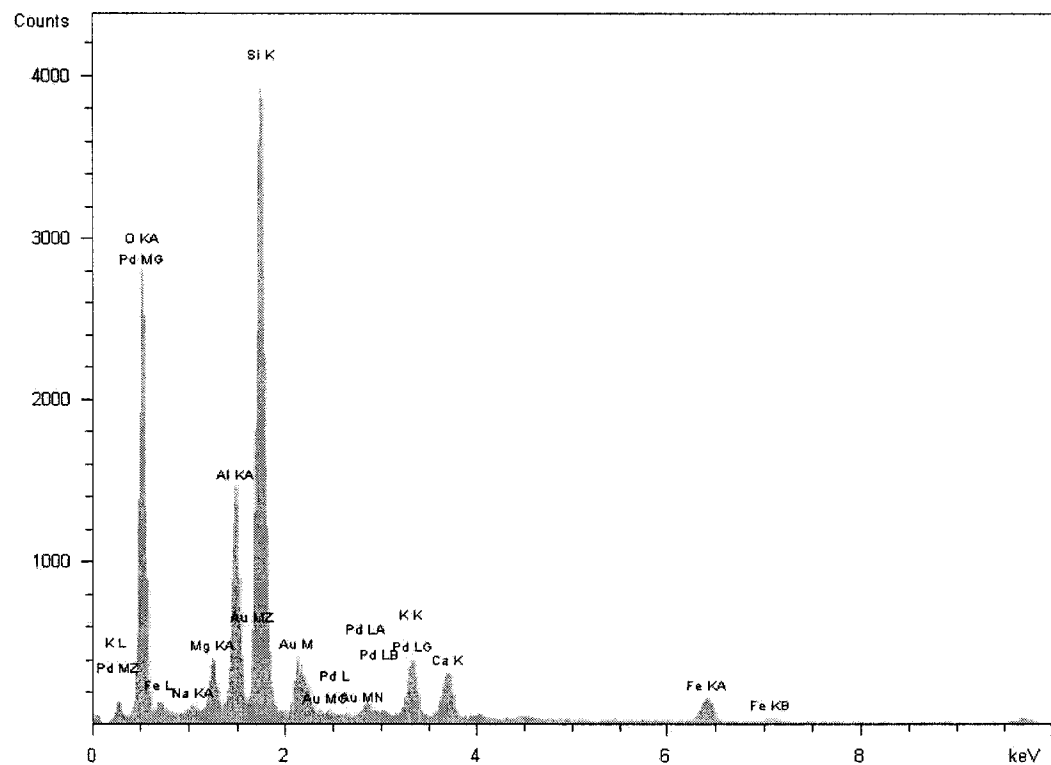


Figure 77 – EDS graph for sample FL-00-9P-III-30 Photo 1, plate 3b.

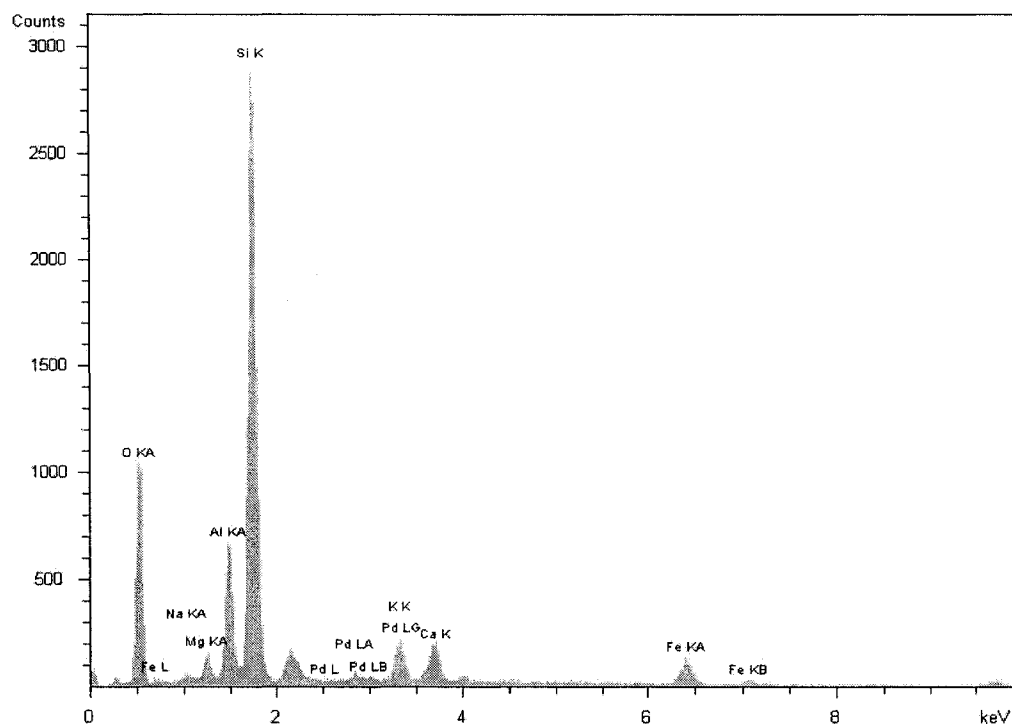


Figure 78 – EDS graph for sample FL-00-9P-III-30 Photo 5, plate 3c.

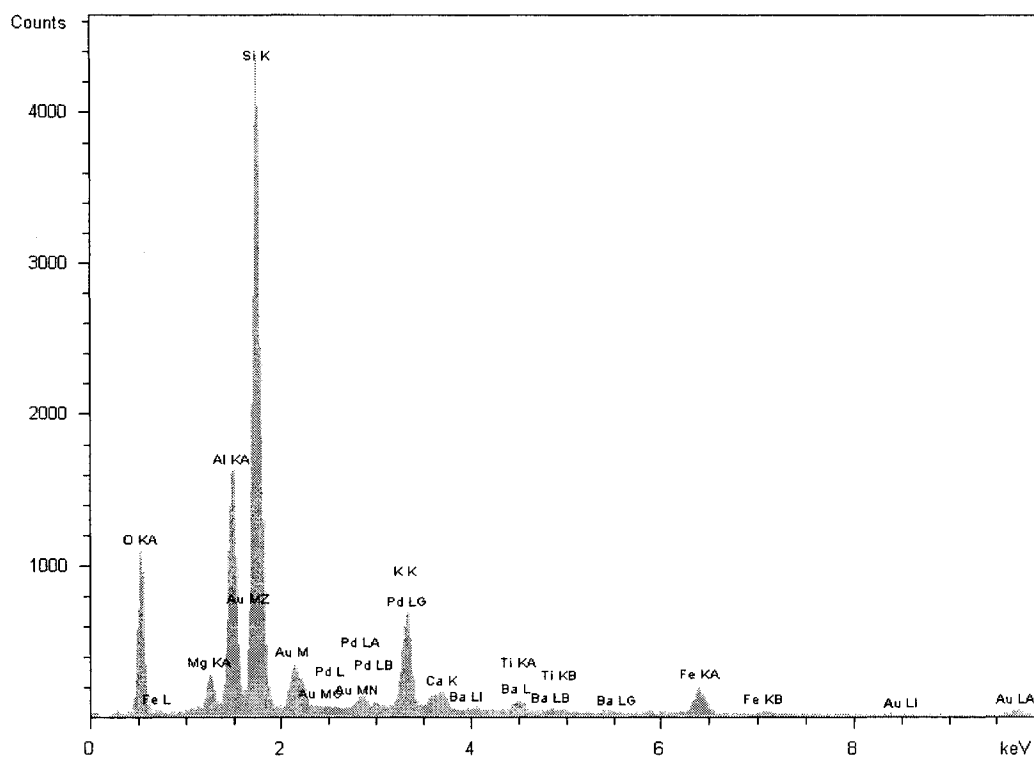


Figure 79 – EDS graph for sample FL-00-9P-IV-10 Photo 3, plate 3d.

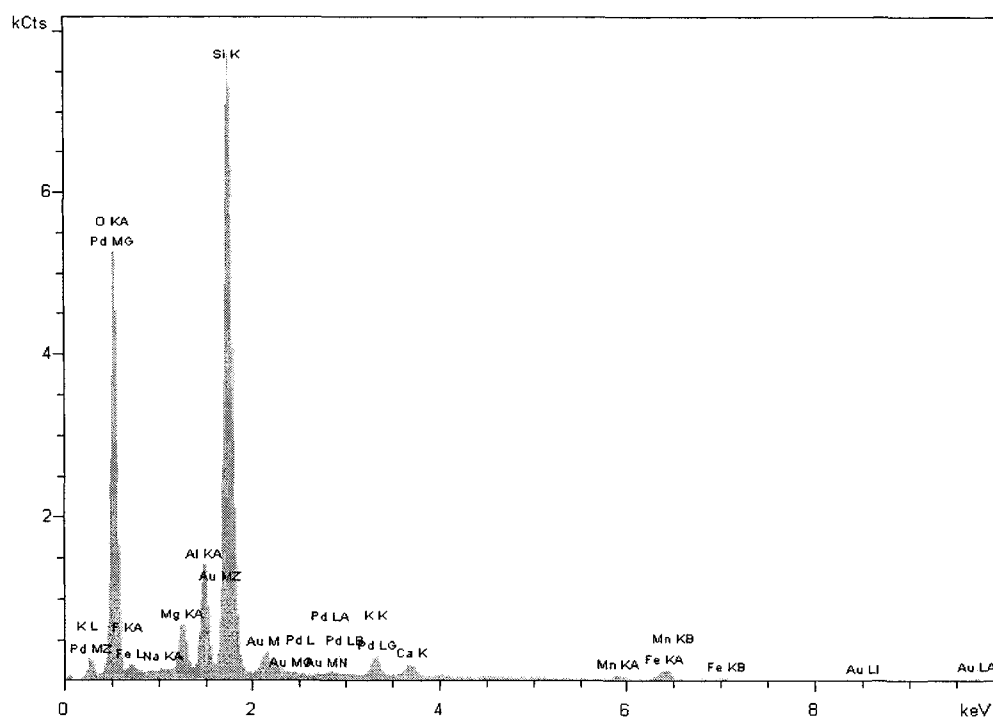


Figure 80 – EDS graph for sample FL-00-9P-IV-10 Photo 6, plate 3e.

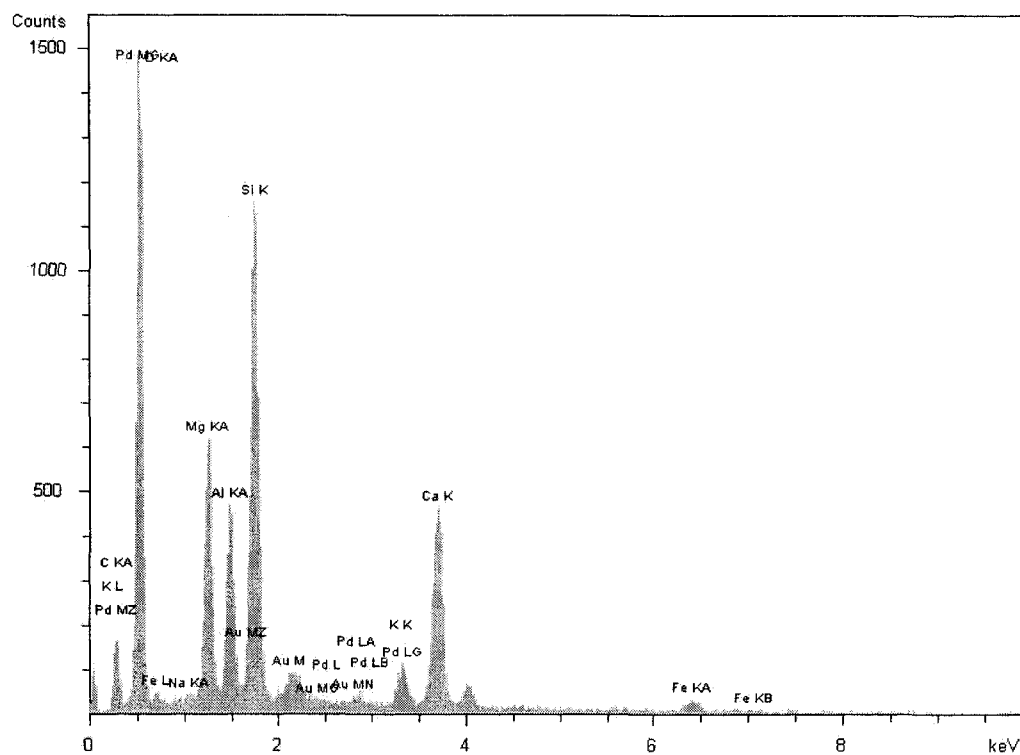


Figure 81 – EDS graph for sample FL-00-9P-V-10B Photo 5, plate 3f.

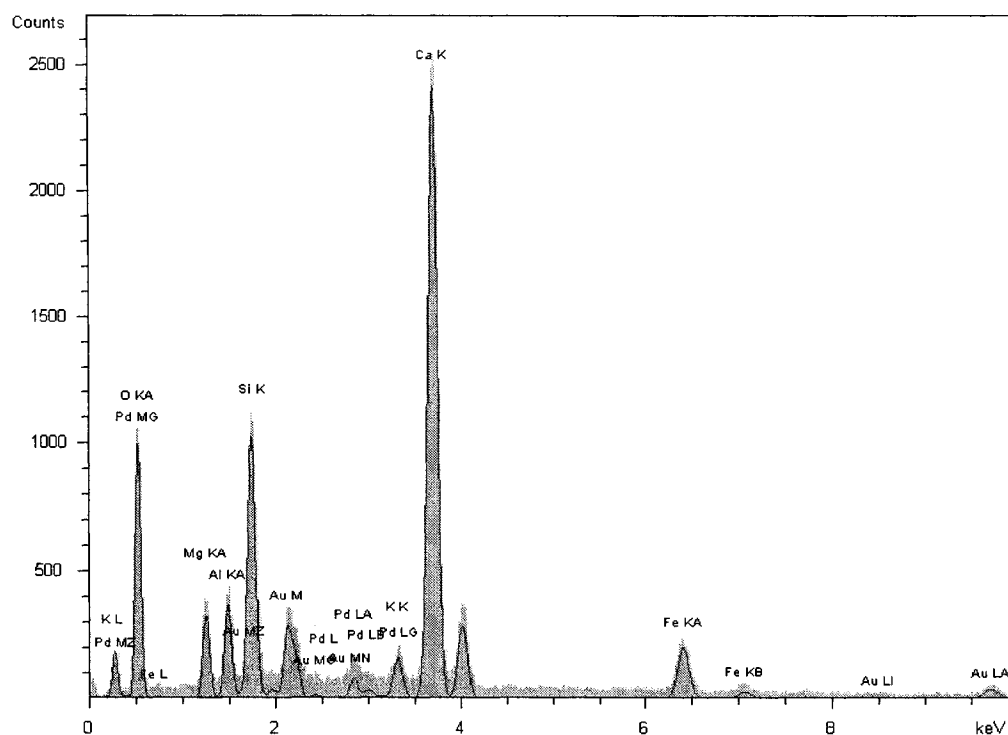


Figure 82 – EDS graph for sample FL-00-9P-V-120 Calcite1, plate 4a.

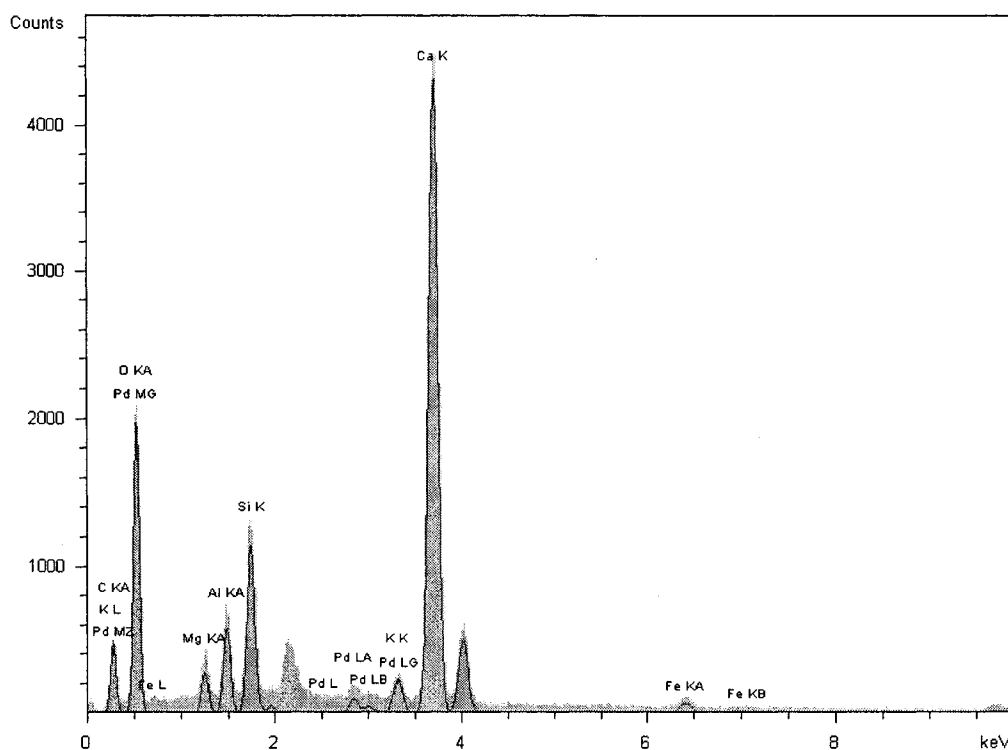


Figure 83 – EDS graph for sample FL-00-9P-V-120 Cal4, plate 4b.

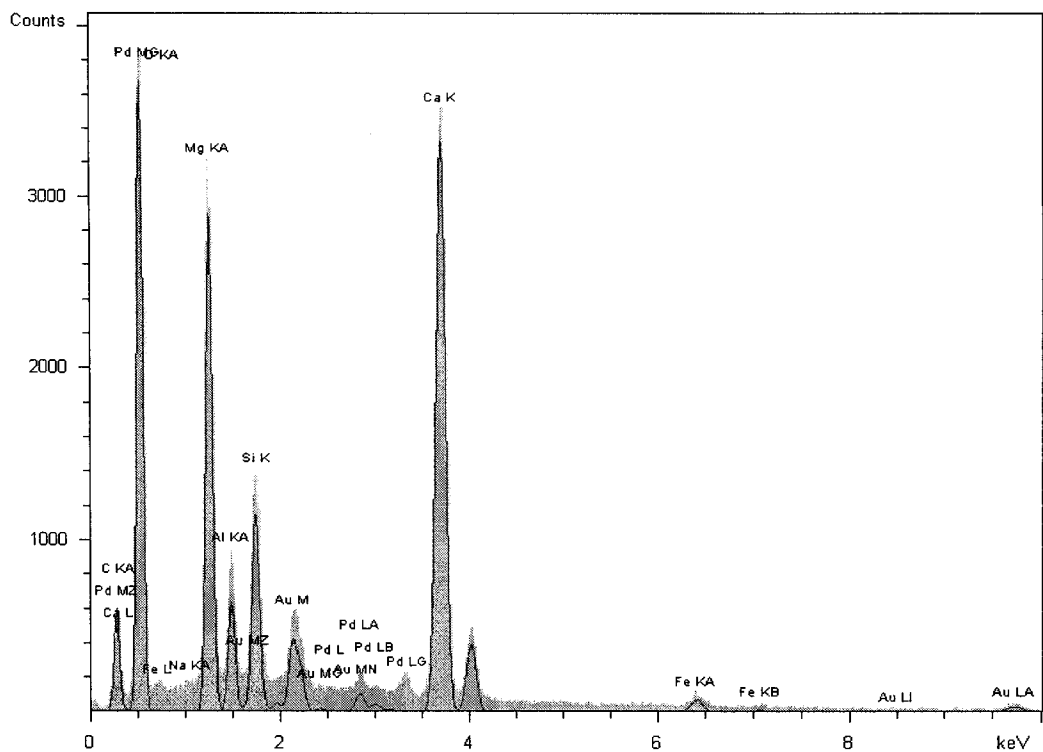


Figure 84 – EDS graph for sample FL-00-9P-V-120 Dolomite, plate 4c.

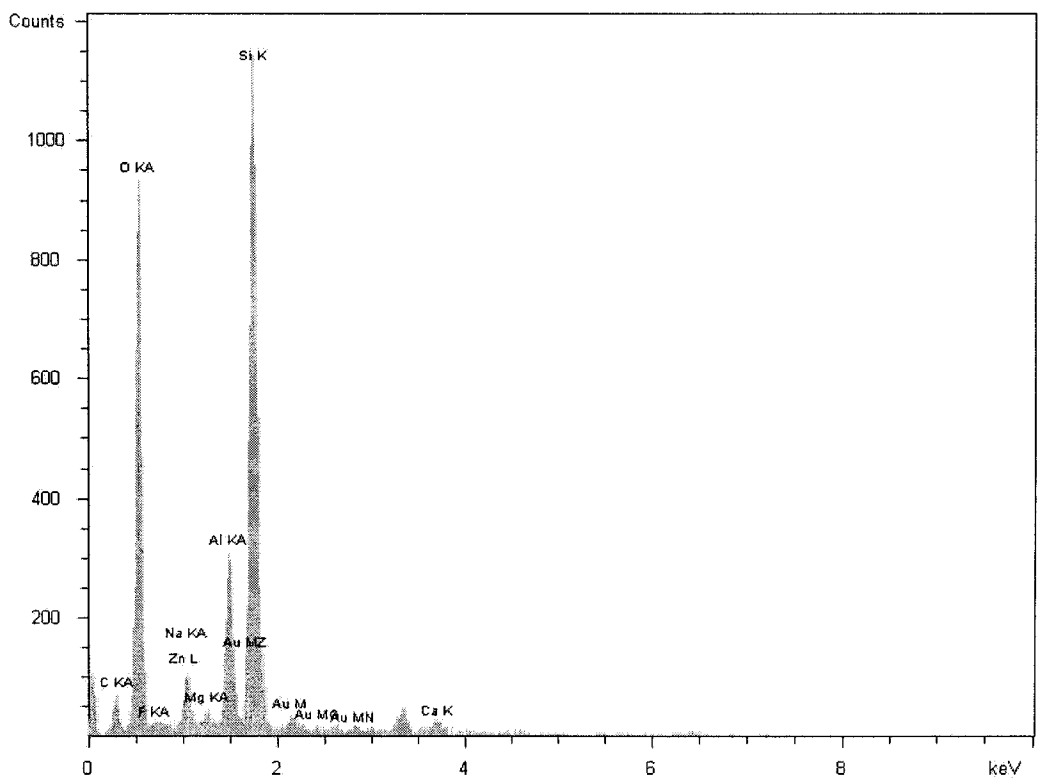


Figure 85 – EDS graph for sample FL-03-16K-II-145 Photo 1, plate 4d.

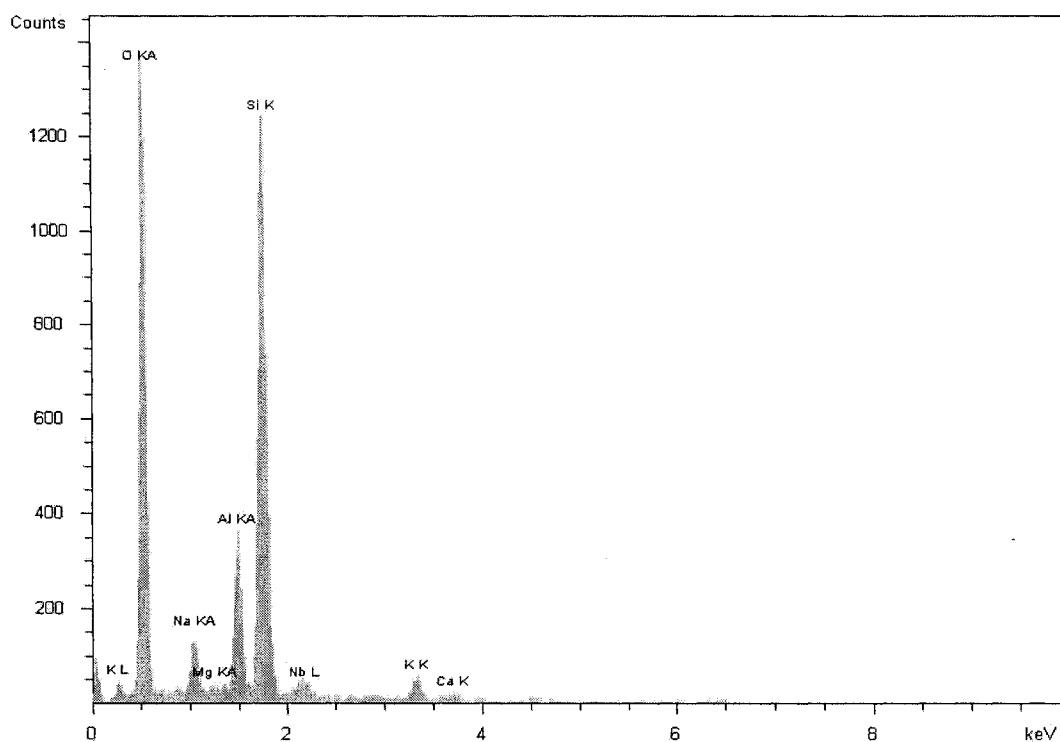


Figure 86 – EDS graph for sample FL-03-16K-II-145 Photo 8, plate 4e.

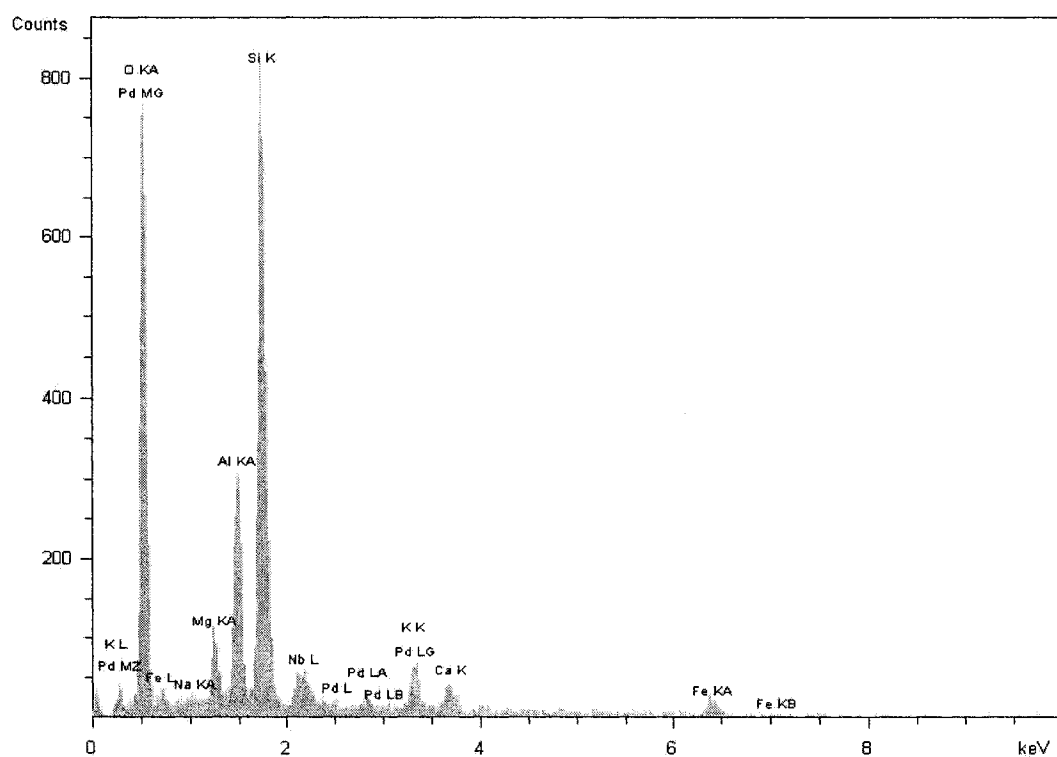


Figure 87 – EDS graph for sample FL-03-15K-V-161 Photo 1, plate 4f.

Appendix F:

Data CD – Contents and Descriptions

The figures presented within this dissertation represent only a portion of the total data collected in the course of this research. In the course of my research a total of ~4.25GB of data were collected in ~2600 files. This makes printing all the data tables cost prohibitive and impracticable for use by future researchers. This Appendix includes two digital video discs (DVD) that contain all the data collected on Flathead Lake regardless if it was utilized in these chapters. The files contain both the raw and processed data. Most data files can be opened or imported into a spreadsheet program like Microsoft Excel. Other files are graphics that require programs such as Golden Software Grapher, Adobe Photoshop or Illustrator.

On Disc 1 are all core data is contained in folders organized by core number. Within the core folders are sub-folders for each of the proxy data group. The Data Master folder contains data summaries and/or files that compare multiple proxies. One spreadsheet file labeled '*core number*-Master' or '*core number*-Master-Calculations' contains the summary of all proxies for each interval analyzed and is the file that is linked to most of the graphic files.

On Disc 2 are all the piston core photos under folders for the coring year. One folder contains all the SEM and EDS images and data files. Another folder contains data generally related to Flathead Lake or the basin.

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