

Be DYNAMICS DURING PEDOGENESIS AND EROSION – A COMPARISON OF METEROIC
 $^{10}\text{Be}/^9\text{Be}$ RATIOS AND IN SITU ^{10}Be – DETERMINED EROSION RATES

A Thesis Proposal Presented

by

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Abstract.

^{10}Be (half life = 1.39×10^6 years) is a cosmogenic radionuclide (Korschinek et al. 2010). Meteoric ^{10}Be ($^{10}\text{Be}_{\text{met}}$) is generated in the atmosphere and deposited on surfaces (Lal and Peters 1967). Despite its use as a tracer of surface processes, there are observations that $^{10}\text{Be}_{\text{met}}$ can remobilize (Graly et al. 2010). In contrast, in situ ^{10}Be ($^{10}\text{Be}_{\text{is}}$), forms inside mineral grains and is not susceptible to remobilization (Bierman 1994). However, analyzing $^{10}\text{Be}_{\text{is}}$ is time-consuming, expensive, and only possible where sand-size grains of quartz are available. The stable isotope ^9Be is present in grain coatings and may have similar reactivity to $^{10}\text{Be}_{\text{met}}$. If so, the $^{10}\text{Be}_{\text{met}}/^9\text{Be}$ ratio of a sample remains fixed, even if remobilization occurs (Von Blanckenburg et al. 2012). I will use several extraction techniques to understand the distribution of ^9Be in samples that have already been analyzed for $^{10}\text{Be}_{\text{met}}$ and $^{10}\text{Be}_{\text{is}}$. By removing the entire grain coating, I can calculate $^{10}\text{Be}_{\text{met}}/^9\text{Be}$ ratios for samples from a variety of tectonic and climatic environments. If systemic variation between ratios and $^{10}\text{Be}_{\text{is}}$ -derived erosion rates exists, $^{10}\text{Be}_{\text{met}}/^9\text{Be}$ could be used as a complement to $^{10}\text{Be}_{\text{is}}$ in determinations of erosion rates, particularly in areas where quartz is not available.

Introduction. Measurements of ^{10}Be produced in rock and soil (in situ ^{10}Be , hereafter $^{10}\text{Be}_{\text{is}}$) have revolutionized our understanding of erosion rates at a drainage basin scale by providing a quantitative method of measuring average denudation rates over millennia (Bierman 1994, Portenga and Bierman 2011). However, due to methodological constraints, $^{10}\text{Be}_{\text{is}}$ measurements are limited to areas where quartz-bearing rocks crop out and where sand-size quartz grains can be isolated (Lal and Peters 1967, Nishiizumi et al. 1986). Over the past 30 years, researchers have also measured ^{10}Be that was formed in the atmosphere, deposited onto exposed surfaces, and remained sorbed onto grains (Monaghan et al. 1986, McKean et al. 1993, Reusser and Bierman 2010). Such meteoric ^{10}Be ($^{10}\text{Be}_{\text{met}}$) could be a useful tracer of sediment movement and basin scale erosion rates (Pavich et al. 1984, Graly et al. 2010). However, because pedogenic processes and grain size strongly influence $^{10}\text{Be}_{\text{met}}$ concentrations on grain surfaces, interpretation of the data is not straightforward (Von Blanckenburg 2005, Graly et al. 2010). Some (Barg et al. 1997, Von Blanckenburg et al. 2012, Bacon et al. 2012) have proposed to normalize $^{10}\text{Be}_{\text{met}}$ measurements using the stable nuclide, ^9Be , as a way to improve interpretations. Since pedogenic processes are unlikely to discriminate between Be isotopes, the

$^{10}\text{Be}_{\text{met}}/^{9}\text{Be}$ ratio should only be influenced by erosion rate, $^{10}\text{Be}_{\text{met}}$ deposition rate, and radioactive decay (Veselý et al. 2002).

This project proposes to extract sequentially ^{9}Be from soils and sediments in order to quantify the amount and distribution of ^{9}Be in grain coatings and mineral matrixes. I will also do a total extraction of ^{9}Be held in grain coatings in order to calculate the $^{10}\text{Be}_{\text{met}}/^{9}\text{Be}$ ratio and by comparison to existing data, determine whether it is a useful proxy for the erosion rate of drainage basins.

Beryllium can exist on sediment and soil grains in several species, which are either adsorbed to the surface of grains (exchangeable), adsorbed into the structure of mineral phases (oxy-hydroxides, carbonates, silicate minerals), or complexed with organic material. Tessier et al. (1979) outline a sequential extraction procedure that partitions particulate trace metals in to five fractions: exchangeable, bound to carbonate, bound to Fe-Mn oxides, bound to organic matter, and residual. Wittmann et al. (2012) optimized the Tessier et al. (1979) procedure specifically for Be extraction from fluvial sediments. I will further optimize this sequential extraction method for soils, fluvial sediments, and lake core sediments. Analyzing the quantity of ^{9}Be in each of the sequential extraction fractions will allow for: 1. better understanding the mechanisms of pedogenesis that allow Be to be mobile in natural systems and 2. better understanding of which fractions (or combinations of fractions) contain ^{9}Be concentrations that, when used to normalize $^{10}\text{Be}_{\text{met}}$ data, correlate to previously measured $^{10}\text{Be}_{\text{is}}$ soil profiles and $^{10}\text{Be}_{\text{is}}$ -derived erosion rates. Because $^{10}\text{Be}_{\text{is}}$ extractions are expensive, time consuming and can only be performed if sand sized quartz is present, normalizing $^{10}\text{Be}_{\text{met}}$ to ^{9}Be could verify $^{10}\text{Be}_{\text{met}}/^{9}\text{Be}$ ratios as an alternative long-term erosion rate monitoring tool that could be used worldwide.

Background.

Natural distribution of Be. Beryllium has three naturally occurring isotopes – ^{10}Be with a half-life of 1.39×10^6 yrs (Korschinek et al. 2010), ^7Be with a half-life of 53 days, and ^9Be , the only stable isotope and the only isotope with greater than trace concentrations. ^{10}Be has proven very useful for applications in geomorphology and climatology. Greater than 99% of the ^{10}Be in natural systems is formed in the atmosphere when neutrons (secondary cosmic rays) react with nitrogen or oxygen (Gosse and Phillips 2001). After production, this $^{10}\text{Be}_{\text{met}}$ adheres to aerosols in the atmosphere and is deposited onto the landscape, where, because of its affinity to solids, it accumulates in soils and on sediments (Lal and Peters 1967, Pavich et al. 1986, Monaghan et al. 1986). However, because $^{10}\text{Be}_{\text{met}}$ only adheres to the surface of grains, it is subject to redistribution (or in extremely weathered environments, removal) *via* eluvial and illuvial processes that are independent of physical erosive processes (Figure 1; Takahashi et al. 1998, Graly et al. 2010, Bacon et al. 2012). ^{10}Be is also formed inside mineral grains from interactions between cosmic rays and ^{16}O atoms (Nishiizumi et al. 1986). This $^{10}\text{Be}_{\text{is}}$ accounts for less than 1% of the total ^{10}Be and has been shown to reflect basin-scale erosion rates (Granger et al. 1996, Bierman and Steig 1996) and the erosion rate of outcrops (Nishiizumi et al. 1986, Bierman et al. 2002, Bierman and Nichols 2004).

Nearly all Be in soils and sediment is the stable isotope ^9Be (Willenbring and von Blanckenburg 2009, Wittmann et al. 2012). Beryllium concentrations generally range from 1-15 ppm in upper crust materials, but an initiative by the US Department of Health and Human Services to document trace element concentrations in surficial materials found that ^9Be concentrations of soils (average US concentration of 0.6 ppm), surface water (average US

concentration of 0.24 ppm), and air (average US concentration of 0.03 ng/m³) are much lower than bedrock (Smith et al. 2002). Determining the distribution of ⁹Be in grain coatings by sequential extraction will help understand the reactivity of this trace metal.

Variables that influence Be phase and mobility in grain coatings. Solid-state materials are most reactive at their exposed surfaces, where electronic imbalances encourage red-ox chemical reactions. As a result, smaller grains with a high surface to volume ratio will have the highest capacity to sorb red-ox active metals such as Be (Zumdahl and Zumdahl 2010). Many studies (Graly et al. 2010, Bacon et al. 2012, Reusser and Bierman 2010) show ¹⁰Be_{met} profiles with a mid-depth maximum concentration – a trend that could be explained by a decrease in grain size or a Be affinity for kaolinite and iron/aluminum oxides concentrated in soil B horizons. If ¹⁰Be_{met} and ⁹Be display the same reactivity in pedogenic processes, we would expect grain size and the reactivity of the grain coating to influence ¹⁰Be_{met} and ⁹Be concentrations similarly. A recent study measured a ⁹Be soil profile and corresponding ¹⁰Be_{met} profile, but did not observe a mid-depth concentration peak for ⁹Be, see Figure 2 (Bacon et al. 2012). Although this observation is contrary to the assumption that weathered ⁹Be and ¹⁰Be_{met} respond similarly to post-depositional processes, Bacon et al. extracted ⁹Be with hydroxylamine hydrochloride – a reagent that would likely leave at least a portion of the organic phase coated to the grain – while the ¹⁰Be_{met} was extracted by the Stone's 1988 method of KHF₂ total fusion. It is possible that if Bacon et al. (2012) had extracted ⁹Be and ¹⁰Be_{met} similarly, the depth profiles would be more similar.

The pH of natural environments strongly influences the likelihood that Be will be remobilized, or even removed, from the studied system. In very acidic environments (pH < 4.1), soil solutions are under-saturated with respect to gibbsite Al(OH)₃, and Al will be most stable in

the soluble ion Al^{3+} (Bacon et al. 2012, Figure 3). This Al^{3+} is free to compete with other metals for exchange sites, potentially becoming the principal sorbate on negatively charged surfaces (called the exchangeable phase of the grain coating). Acidic conditions thus encourage Be^{2+} to desorb from the exchangeable phase of the sediment (Willenbring et al. 2009).

Above pH 5, organic ligands can remain deprotonated and able to form organometallic complexes with available cations. Organic-rich soils in strongly acidic to neutral conditions can decrease Be mobility by sequestering Be in organometallic Be^{2+} complexes that have a strong affinity for positively charged Fe-oxy-hydroxides, Al hydroxides, and clay minerals (Willenbring et al. 2009). Further, Taskahashi et al. (1998) found that in soils with pH between 3 and 11, nearly all Be takes the form of the hydroxylated species ($\text{Be}(\text{OH})_x^{-n}$); see Figure 4. Similar to Be^{2+} organic complexes, $\text{Be}(\text{OH})_x^{-n}$ humate complexes readily adsorb onto positively charged crystalline oxide or amorphous oxy-hydrate surfaces. Consequently, Takahashi et al. (1998) observed that when dissolved Be^{2+} (pH 7.5) was mixed with humic acids in the presence of kaolinite, over 70% of Be was adsorbed to the kaolinite in the form of hydroxylated Be humate complexes. This research indicates that if significant concentrations of humic acid are present, Be will remain adsorbed to the crystalline oxide or amorphous oxy-hydrate surfaces, even at low pH.

⁹Be dynamics in the biosphere – a closed system? Although studies of rock, soil, water, and aerosols indicate that significant amounts of ⁹Be are unlikely to be added or removed from the soil column (and can thus be considered a closed system on the millennial timescales used to study pedogenesis and erosion), the degree to which Be cycles through the biosphere is not well constrained (Kabata-Pendias and Szteke 2015, Veselý et al. 2002). The similar reactivity of Be and other common biologically relevant elements such as Mg, Ca, and Al has been used to

explain why Be is incorporated into organic materials. Like Mg and Ca, Be occurs naturally in the +2 oxidation state, and similar to Al^{3+} , Be^{2+} has a high charge to ionic radius ratio. These similar physical characteristics allow for Be to compete with Mg^{2+} , Ca^{2+} , and Al^{3+} in plant nutrient cycling mechanisms (Willenbring et al. 2009, Metcalfe and Little 2007).

Most studies show that unlike heavy metals such as Pb, Be does not bioaccumulate significantly (Kabata-Pendias and Szteke 2015). Meehan and Smythe (1967) found that the overall uptake of Be by living organisms is low, even in acidic soils where Be may be more mobile. Other research, however, shows that some plants absorb significant quantities of Be from the soil (Vesely et al. 2002). Conyers (2014) reported meteoric Be concentrations of the trees and surrounding soils in Martell Experimental Forest, Indiana and found that Hickory wood samples contained ~ 0.38 ppm ^{10}Be by dry weight in wood, and ~ 2.0 ppm ^{10}Be in fallen leaves, while the concentration of ^{10}Be in the surrounding soil was measured as less than 0.2 ppm. Although these trees showed ^{10}Be concentrations an order of magnitude higher than the surrounding soils, ^{10}Be appeared to be equally bioavailable as the ^9Be , with $^{10}\text{Be}/^9\text{Be}$ ratios of all tree leaves tested within an order of magnitude of the $^{10}\text{Be}/^9\text{Be}$ ratio of soil samples along a 100 cm depth profile (Conyers 2014). This study is encouraging because it suggests that while Be can be mobilized via biological pathways, the $^{10}\text{Be}/^9\text{Be}$ ratio remains within an order of magnitude.

While there is no agreement on the degree to which the biosphere cycles Be, the high concentrations of Be measured in coal seem to indicate that bioaccumulation could be significant. Beryllium concentrations in coal are commonly measured between 10 and 330 ppm, although concentrations as high as 2000 ppm have also been observed (Vesely et al. 2002). As there are regions that have coal deposits that erode with fluvial sediment or are incorporated into the soil profile, the added contribution of Be from coal could act as an additional source of ^9Be .

In these settings, precautions will be necessary to either measure or remove the coal component of samples.

Initial Sample Selection. The first phase of this project will analyze a set of fluvial sands from the Susquehanna River Watershed ($10^{10}\text{Be}_{\text{met}}$ and $^{10}\text{Be}_{\text{is}}$ sample pairs), a set of acidic peat soil samples along a depth profile in Scotland ($10^{10}\text{Be}_{\text{met}}$ and $^{10}\text{Be}_{\text{is}}$ sample pairs), and a set of homogenized varve samples from a series of stratigraphic sections in the Connecticut River Valley (10 samples, only measured for $^{10}\text{Be}_{\text{met}}$) (Reusser and Bierman 2010, DeJong et al. 2012, Fulop et al. 2015). The varve samples can be sub-divided into two sets, one that was accumulated relatively distal to the retreating ice sheet, and another that was accumulated proximal to the ice sheet. The proglacial lake sediment data sets contrast conditions of incipient soil formation several thousand years after glaciation with a more barren, recently glaciated landscape. These soil, sand, and varve samples have been selected as primary sample sets because they represent a heterogeneous set of samples that have differences in variables that influence ^{10}Be and ^9Be concentration, such as acidity, grain size and biological activity.

Analysis of these three sample sets attempt to probe questions related to this study's main objectives. 1. Are $^{10}\text{Be}_{\text{met}}/^9\text{Be}$ ratios useful as a proxy for the previously determined $^{10}\text{Be}_{\text{is}}$ derived erosion rates in soils and fluvial sands? 2. Does the ratio of $^{10}\text{Be}_{\text{met}}/^9\text{Be}$ align with previously measured $^{10}\text{Be}_{\text{is}}$ along depth profiles of soils? 3. Overall, what does the concentration and phase of ^9Be in grain coatings of soils, fluvial sediments, and lake sediments tell us about pedogenesis and Be dynamics in natural settings?

For the second phase of this project, a large number of samples will be analyzed by an extraction method that removes the entire grain coating but does not attack the mineral matrix, such as leaching in HCl or HNO_3 . Ten samples from each location will also be measured by

sequential extraction. These samples will provide more data to support conclusions drawn in the first phase of the project, and will cover a gradient of climates and tectonic regimes (see Table 1. for a list and description of sample locations.) Providing data that shows how $^{10}\text{Be}_{\text{met}}/^9\text{Be}$ ratios behave across climates and tectonic regimes will test the validity of using $^{10}\text{Be}_{\text{met}}/^9\text{Be}$ ratios as an erosion rate proxy in more extreme end members of erosion and climactic environments.

Hypothesis. Relatively few studies of ^9Be sequential extractions from soils and sediments have been performed (Barg et al. 1997, Bacon et al. 2012, Wittmann et al. 2012), so it is difficult to predict which fractions of sequential Be extraction will have the highest ^9Be concentrations. Barg et al. (1997) and Wittmann et al. (2012) show that Be accumulates in organic-rich and clay-rich layers of soils. I therefore hypothesize that the sequential extraction fractions that selectively dissolve organic and exchangeable phases will liberate the largest quantity of Be. However, in samples with significant amounts of humic acids, I hypothesize that the crystalline oxide and amorphous oxide-bound fractions will contain significant amount of ^9Be (Taskahashi et al. 1998). Because the total grain coating is extracted for $^{10}\text{Be}_{\text{met}}$ analysis, I hypothesize that the ^9Be from the total grain coating will result in the most meaningful relationship between ^9Be , $^{10}\text{Be}_{\text{met}}$, and long-term erosion rates. I hypothesize that there will be an increased concentration of ^9Be in grain coatings in the distal glacial lake sediments than the proximal sediments because ^9Be will have become more mobile during pedogenesis in the time after glaciation.

Many published reports show results that indicate the $^{10}\text{Be}_{\text{met}}/^9\text{Be}$ ratio normalizes $^{10}\text{Be}_{\text{met}}$ data to account for grain size effects, $^{10}\text{Be}_{\text{met}}$ remobilizing and/or $^{10}\text{Be}_{\text{met}}$ leaching (Merrill et al. 1959, Barg et al. 1997, Bacon et al. 2012, Conyers 2014, Von Blanckenburg et al. 2012, Wittmann et al. 2010, Willenbring and von Blanckenburg 2010). Because publications indicate that $^{10}\text{Be}_{\text{met}}/^9\text{Be}$ ratios could be meaningful indicators of erosion, I hypothesize that a $^{10}\text{Be}_{\text{met}}/^9\text{Be}$

ratio that includes the total ^9Be in the outside coating of grains will correlate with erosion rates calculated using $^{10}\text{Be}_{\text{is}}$ data.

Methods.

The initial methods for ^9Be sequential extraction will be adapted from of the sequential extraction published by Wittmann et al. (2012) (see Figure 5. for reagents and targeted fractions). Before running the sequential extraction, I will measure the average grain size for each sample set to ensure that the samples have all been powdered to a similar size. Each sample will result in five fractions that will be analyzed for ^9Be concentration on the UVM Geology Department's JY Horiba Optima Ion Coupled Plasma-Optical Emission Spectrometer (ICP-OES). In addition to the sequential extraction, I will use a mineral acid (HCl or HNO_3) to remove the total grain coating. This will allow me to measure the total ^9Be in the coating that should be liberated during the sequential extraction. Because $^{10}\text{Be}_{\text{met}}$ data are derived from total fusion of grains, the ^9Be in the total grain coating will be most likely to correlate with $^{10}\text{Be}_{\text{met}}$ and $^{10}\text{Be}_{\text{is}}$. I will monitor the silicate concentration of the supernatant in these extractions during measurements on the ICP-OES to ensure that the reagent is not attacking mineral grains.

To test the hypothesis that $^{10}\text{Be}_{\text{met}}$ measurements made in sediment and soil can accurately reflect rates of landscape denudation if native ^9Be is also measured, statistical tests such as ordinary least squares (a simple regression) will be used. Regressions will also be used to assess the influence of climate and tectonic regime on $^{10}\text{Be}_{\text{met}}/^9\text{Be}$ ratios.

Research Plan.

1. Refine a selective sequential extraction procedure for ^9Be that can be used for soil and sediment samples and a total grain coating extraction method that will not attack the mineral

matrix.

2. Measure ^9Be in the extracted fractions and calculate $^{10}\text{Be}_{\text{met}}/^9\text{Be}$ ratios for soil and sediment samples in which $^{10}\text{Be}_{\text{is}}$ and $^{10}\text{Be}_{\text{met}}$ have already been measured.

3. Compare $^{10}\text{Be}_{\text{met}}/^9\text{Be}$ ratios to previously measured $^{10}\text{Be}_{\text{is}}$ data to determine what (if any) systematic variations exist.

4. Compare $^{10}\text{Be}_{\text{met}}/^9\text{Be}$ ratios and the distribution of ^9Be to climate or tectonic regimes to determine what (if any) correlation exists.

Implications. This project combines measurements of $^{10}\text{Be}_{\text{met}}/^9\text{Be}$ ratios, $^{10}\text{Be}_{\text{met}}$ and ^9Be to better understand the meaning of $^{10}\text{Be}_{\text{met}}$ data. If the hypothesis that native ^9Be chemically behaves similarly to meteoric ^{10}Be in weathering and pedogenic reactions is true, the $^{10}\text{Be}_{\text{met}}/^9\text{Be}$ ratio could in theory be used to normalize the $^{10}\text{Be}_{\text{met}}$ nuclide inventory to account for post-deposition chemical processes, thereby unlocking a powerful and easy-to-measure tool for determining rates of erosion using fluvial sediments, ancient soils, or lacustrine sediment cores (Merrill et al. 1959, Bacon et al. 2012, Wittmann et al. 2012). In the processes of extracting ^9Be from these samples, I will learn more about the processes that control Be mobilization in natural settings.

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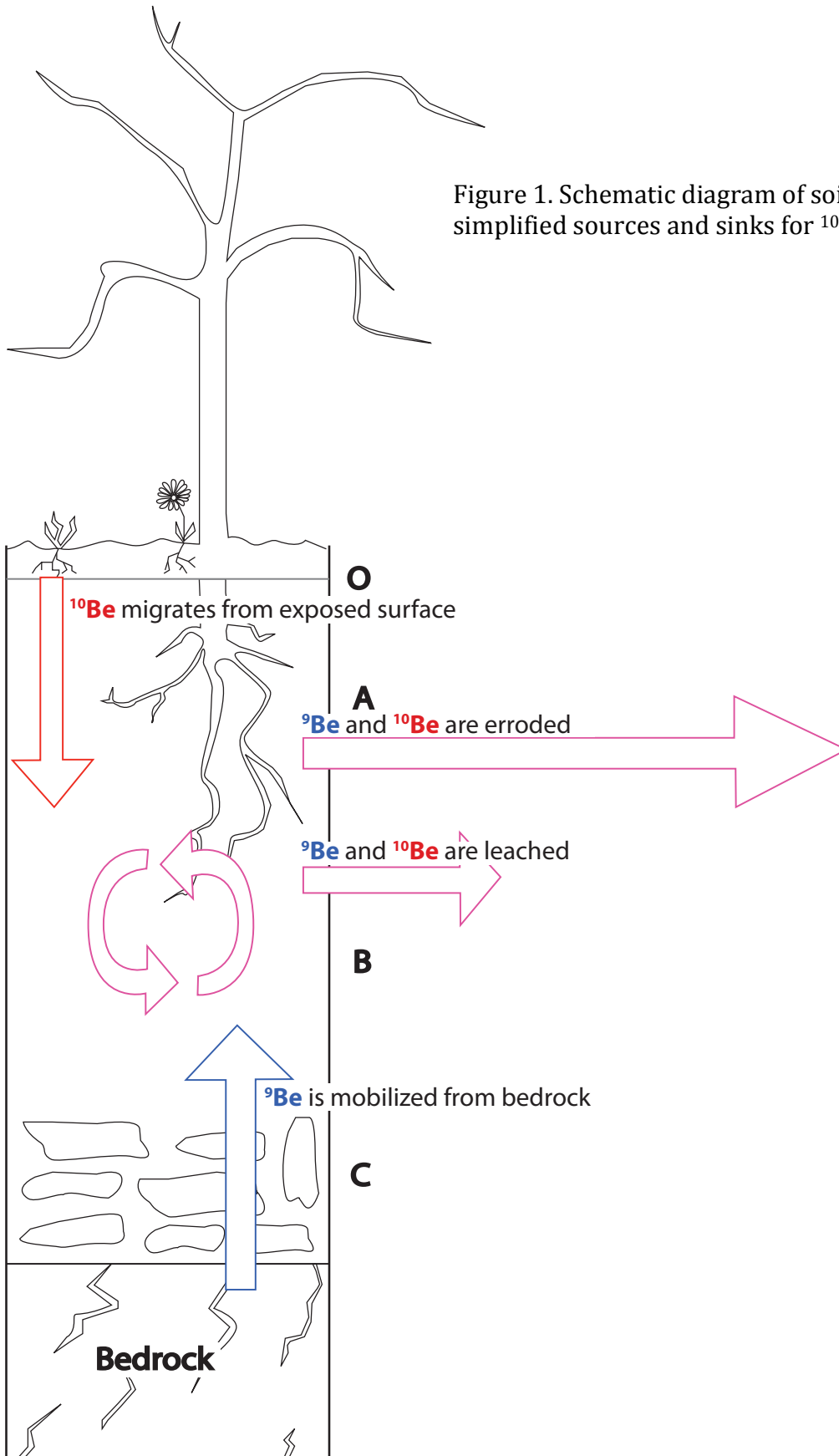
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Figure 1. Schematic diagram of soil profile illustrates simplified sources and sinks for ^{10}Be and ^9Be .



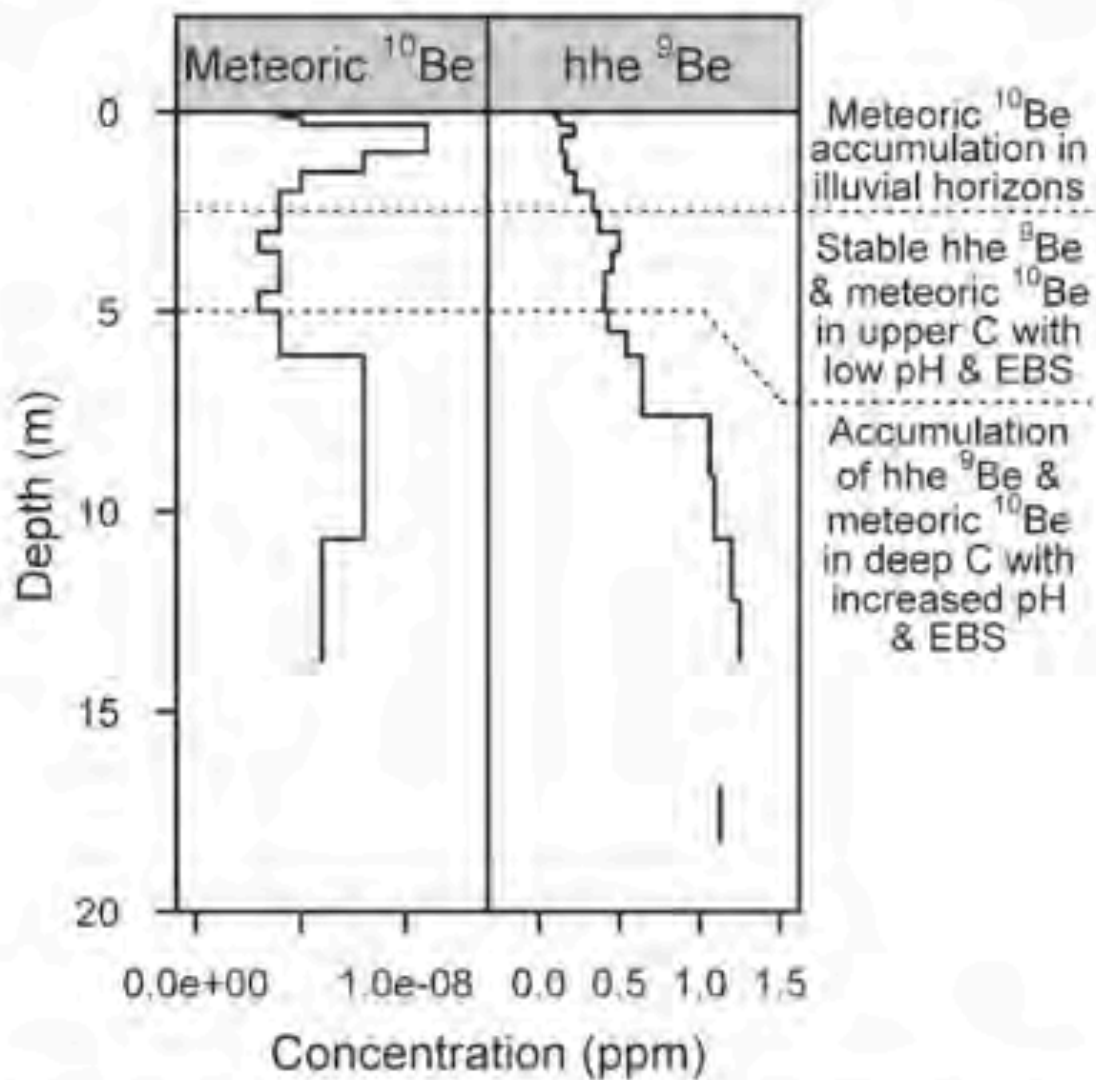


Figure 2. Data from Bacon et al. 2012. Direct observation of ^{10}Be met and hydroxylamine hydrochloride extractable ^9Be in Southern Piedmont Utiisol soil profile. EBS = “effective base saturation.”

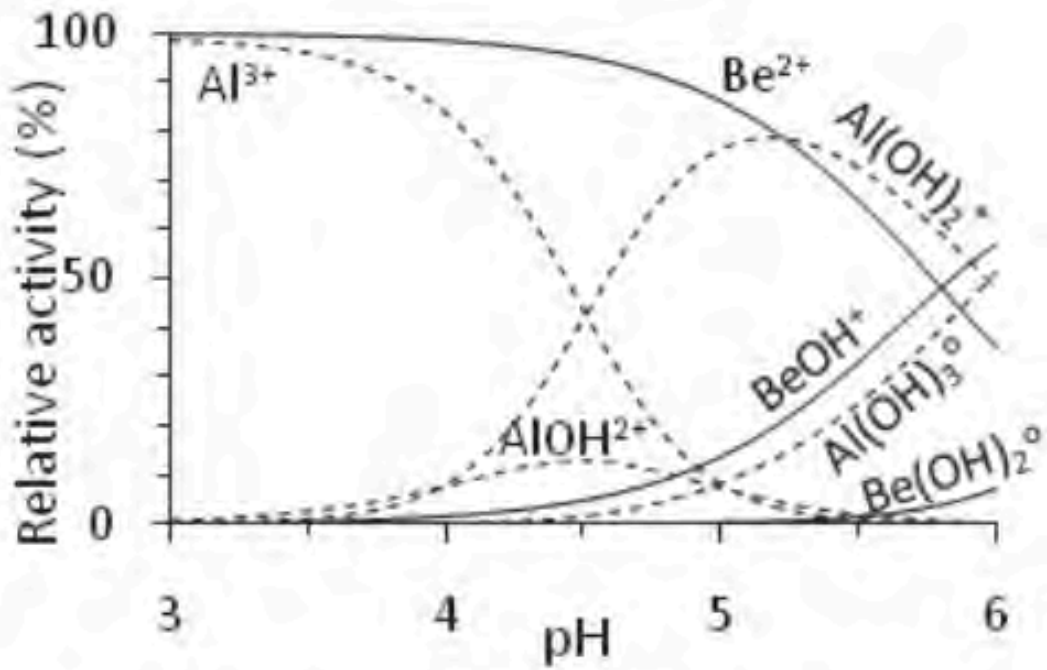


Figure 3. Aluminum and Beryllium speciation in water at varied pH. Figure from Bacon et al. 2012, data from Lindsey 1979, Renders and Anderson 1987, and Sparks 2003.

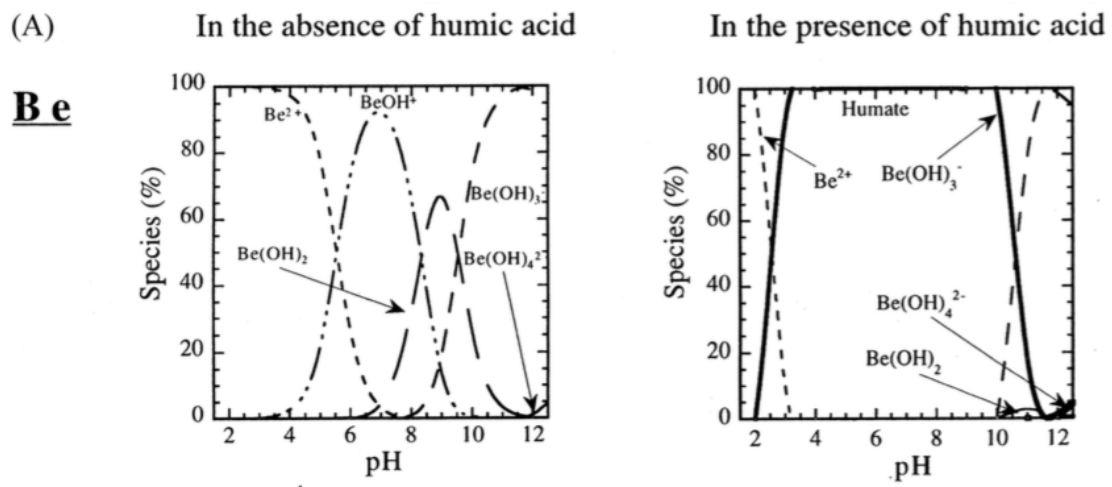


Figure 4. Beryllium speciation in water with and without humic acid. Figure from Takahashi et al. 1998.

Sequential Extractions Method (modified from Wittmann et al. 2012)

If possible, check to see the grain size of powdered sample

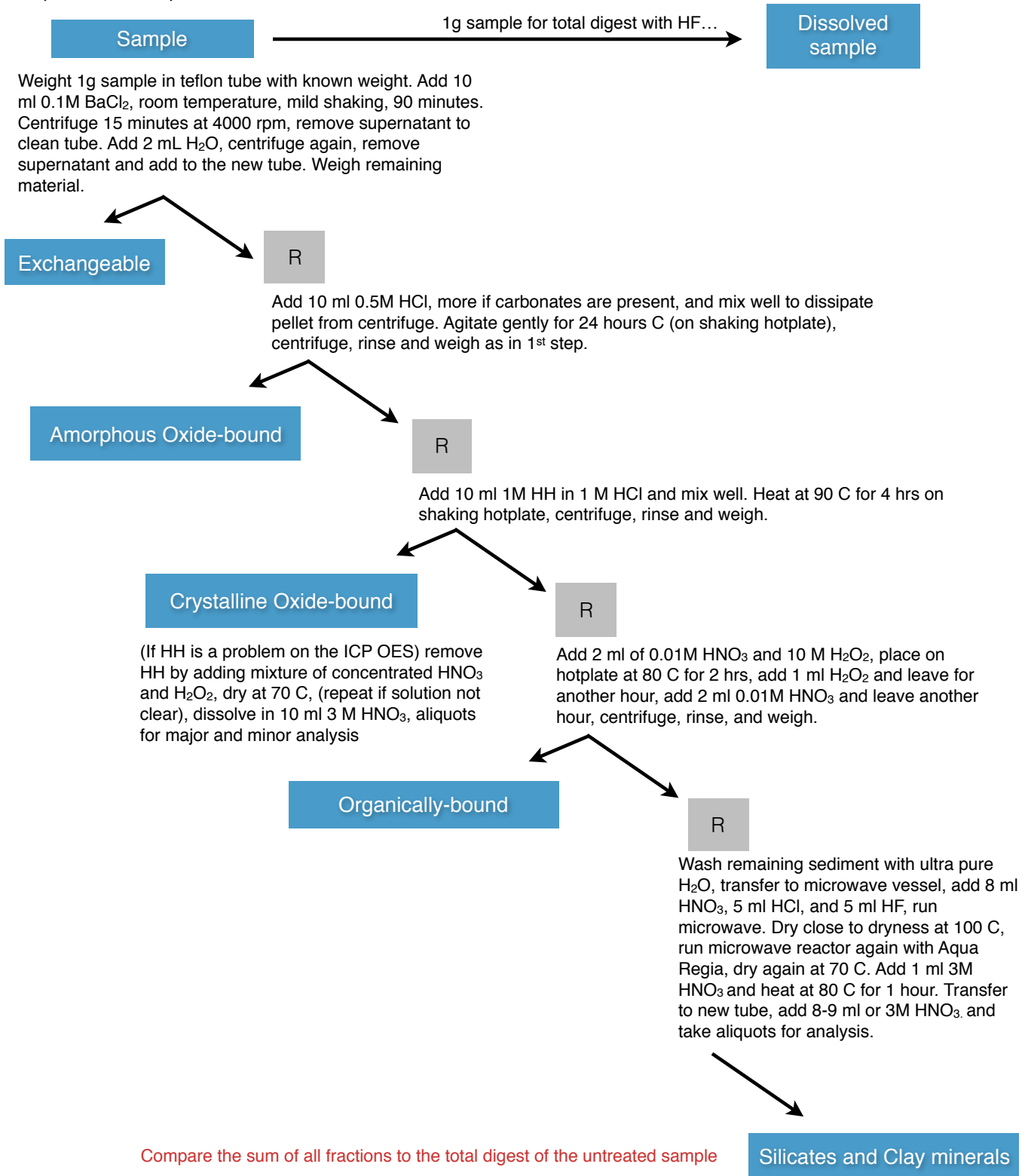


Figure 5. Sequential extraction procedure, as modified from Wittmann et al. 2012.

sample	climate	soil type	tectonic setting	rock type/parent material	precip (mm/yr)	pub sources	In situ met sample pairs	sediment yield data?
Susquehanna River Sands	humid temperate, glaciated	n/a	passive margin	deformed metamorphic rocks, fold and thrust belt with sandstone, shale, and carbonate, far N and W sandstone and shale	800-1300	Joanna Reuter, UVM thesis	17 paired	yes
NAVC varves	glacial and immediately post glacial	n/a	passive margin	metamorphic and igneous intrusive (granites)	no data	NAVC proposal	200+ met only	sort of...varve thickness
Scottish soil profile	humid temperate, glaciated	blanket bog peat over glacial till	passive margin	metamorphic and igneous intrusive	~1100	Fuklop et al 2015	18 paired	n/a
Potomac River Sands	humid temperate, never glaciated	n/a	passive margin	deformed metamorphic rocks, sandstone, shale, and carbonate	890-1320	Trodick 2011	62 met, 8 paired	yes
Barron River Sands (NE Australia)	humid, tropical, never glaciated	n/a	passive margin	granitic and biogenetic carbonate	900-2500	Nichols et al 2014	15 paired	modeled
Georges River Sands (SE Australia)	humid temperate, never glaciated	n/a	passive margin	trassic sandstone, granodiorite	800-1300	not published	9 paired	modeled
China, 3 Rivers Region Sands	tropical, never glaciated	n/a	tectonically active	lightly metamorphosed granite and sedimentary	500-1000	not published	>120 paired	some
Nambian River Sands	sub-humid to hyperarid, tropical, never glaciated	n/a	passive margin	granitic and gneissic	25-400	Some in Bierman and Caffee, 2001	12 paired	no
Waipolia River Sands	temperate, humid, never glaciated	n/a	tectonically active	carbonate bearing silt and sandstone	1000-1500	Reusser and Bierman 2010, not all data published	90 met; 18 paired	yes
Waipolia River Soil Pit	temperate, humid, never glaciated	pumice and podzol	tectonically active	river terrace sand and silt derived from carbonate bearing silt and sandstone	1000-1500	Reusser et al 2010	13 met only	n/a
Great Smokey Mountains soil profiles	temperate, never glaciated	Inceptisols	passive margin	meta-siltstone, metamorphosed conglomeratic sandstone, carbonates and gneiss	1400-2300	Jungers et al, 2009	59 paired	n/a

Table 1. List of sample locations with pairs of $^{10}\text{Be}_s$ and $^{10}\text{Be}_{\text{met}}$ data. These samples could provide samples for analysis in location that span climatic and tectonic regimes.

Timeline

