



Research paper

An approach for optimizing *in situ* cosmogenic ^{10}Be sample preparationLee B. Corbett ^{a,*}, Paul R. Bierman ^a, Dylan H. Rood ^{b, c}^a Department of Geology and Rubenstein School of the Environment and Natural Resources, University of Vermont, Burlington, VT, 05405, USA^b Department of Earth Science and Engineering, Imperial College London, South Kensington Campus, London, SW7 2AZ, UK^c Center for Accelerator Mass Spectrometry, Lawrence Livermore National Laboratory, Livermore, CA, 94550, USA

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ABSTRACT

Optimizing sample preparation for the isotopic measurement of ^{10}Be extracted from quartz mineral separates has a direct positive effect on the accuracy and precision of isotopic analysis. Here, we demonstrate the value of tracing Be throughout the extraction process (both after dissolution and after processing), producing pure Be (by optimizing ion exchange chromatography methods and quantifying quartz mineral separate and final Be fraction purity), and minimizing backgrounds (through reducing both laboratory process blanks and ^{10}B isobaric interference). These optimization strategies increase the amount of ^{10}Be available for analysis during accelerator mass spectrometry (AMS), while simultaneously decreasing interference and contamination, and ensuring that sample performance matches standard performance during analysis. After optimization of our laboratory's extraction methodology, $^9\text{Be}^{3+}$ ion beam currents measured during AMS analysis, a metric for sample purity and Be yield through the extraction process, matched the $^9\text{Be}^{3+}$ beam currents of AMS standards analyzed at the same time considering nearly 800 samples. Optimization of laboratory procedures leads to purer samples that perform better, more consistently, and more similarly to standards during AMS analysis, allowing for improved precision and accuracy of measurements used for dating and quantification of Earth surface processes.

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1. Introduction

Measurement of *in situ* produced cosmogenic ^{10}Be in geologic samples provides insight about a wide variety of geologic processes (Bierman and Nichols, 2004; Gosse and Phillips, 2001; Granger et al., 2013; Nishiizumi et al., 1993; von Blanckenburg and Willenbring, 2014). For example, quantifying ^{10}Be concentrations in moraine boulders (Heyman et al., 2011; Phillips et al., 1990) or previously-glaciated bedrock surfaces (Bierman et al., 1999) provides information about past glacial behavior, thus yielding valuable paleoclimatic insight (Balco, 2011; Fabel and Harbor, 1999). Cosmogenic ^{10}Be is useful for measuring displacement rates on fault systems by dating offset landforms (Bierman et al., 1995; Brown et al., 1998; Matmon et al., 2005; Rood et al., 2010). It can also be employed to study landscape erosion rates, both on outcrop scales (Nishiizumi et al., 1991, 1986) and basin scales (Bierman and Steig, 1996; Brown et al., 1995; Granger et al., 1996; von

Blanckenburg, 2005), thereby providing insight about Earth's changing surface (Portenga and Bierman, 2011).

Cosmogenic ^{10}Be forms *in situ* when high-energy cosmic rays bombard rock in the upper-most few meters of Earth's surface (Lal, 1988). In the mineral quartz, ^{10}Be is produced primarily by spallation of oxygen at low rates, on the order of ~ 4 atoms g^{-1} quartz yr^{-1} at sea level and high latitude (Balco et al., 2008; Borchers et al., 2016). Production of ^{10}Be in rock and soil is primarily dependent on latitude and elevation, and effectively ceases if the sample surface is buried to a depth of more than a few meters (for example, by glacial ice, sediment, or soil). ^{10}Be has a half-life of ~ 1.4 million years (Chmeleff et al., 2010; Korschinek et al., 2010; Nishiizumi et al., 2007). Therefore, while concentrations of ^{10}Be initially increase in exposed rock over time, they eventually level off as production, erosion, and decay reach steady state.

Preparing and analyzing a sample for ^{10}Be measurement requires numerous steps. After a sample is collected, the mineral quartz is isolated from the other mineral phases through a series of physical and chemical processes (Kohl and Nishiizumi, 1992). The quartz is then dissolved in the presence of a ^9Be carrier solution, and Be is chemically isolated. To measure ^{10}Be , atoms of this rare

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isotope are counted in relation to the ion current of stable ^9Be via accelerator mass spectrometry, or AMS (Muzikar et al., 2003; Tuniz et al., 1998). Because isotopic fractionation can occur in the AMS, primary standards such as the KNSTD dilution series (Nishiizumi et al., 2007) are analyzed in association with samples. A correction factor for the measured versus the assumed $^{10}\text{Be}/^9\text{Be}$ ratio of primary standards is determined, then applied to normalize ratio measurements for samples analyzed at the same time.

The accuracy of sample measurement is controlled in part by the overall closeness of match between standards and samples. Developing a correction factor from standard $^{10}\text{Be}/^9\text{Be}$ ratios and using it to normalize sample $^{10}\text{Be}/^9\text{Be}$ ratios relies upon the assumption that standards and samples behave similarly during measurement. Important characteristics may include matrix effects (e.g. accessory elements that could interfere with measurement; Hunt et al. (2008), Merchel et al. (2008)), cathode geometry (e.g. depth to the sputtering surface and shape of the surface; Hunt et al. (2007), Rood et al. (2013), Shanks and Freeman (2015)), total mass, and performance during measurement (the ion source yield, or “beam current”, which we measure as the $^9\text{Be}^{3+}$ current but which can alternatively be measured as the $^9\text{Be}^{16}\text{O}^-$ current). The similarity of beam currents between standards and samples across multiple measurement cycles is particularly important (Rood et al., 2014). Additionally, contamination of a sample with the isobar ^{10}B , above the ability of the detector to reject such interference, inhibits reliable detection of ^{10}Be and has the potential to degrade accuracy (Merchel et al., 2012).

The precision of low $^{10}\text{Be}/^9\text{Be}$ samples is primarily controlled by Poisson counting statistics, with greater numbers of ^{10}Be counts yielding more precise analyses. The total number of attainable counts is a product of the ^{10}Be concentration of the material being analyzed, the total mass of the sample, and the AMS total system efficiency (including ionization, transmission, transport, and detection efficiencies), all of which dictate the number of ^{10}Be counts that can be obtained before the sample material is ablated away during sputtering (Rood et al., 2013, 2010). For higher $^{10}\text{Be}/^9\text{Be}$ samples, precision is primarily controlled by the reproducibility (i.e., scatter) of ratio measurements, which is often poorer than that predicted by counting statistics alone (Rood et al., 2013). For these higher $^{10}\text{Be}/^9\text{Be}$ samples that are limited by reproducibility rather than counting statistics, closeness of match to standards can dictate precision in addition to accuracy (Rood et al., 2014). Background levels of ^{10}Be introduced during sample processing also control the precision of measured isotopic ratios, with relatively higher process blanks increasing the uncertainty of sample $^{10}\text{Be}/^9\text{Be}$ ratios especially in samples with little ^{10}Be , because background uncertainties are typically added in quadrature.

There are several reasons why it is advantageous to optimize the preparation of samples for ^{10}Be isotopic analysis. Ensuring that sample performance matches standard performance during AMS analysis likely increases the accuracy of sample measurements, a prerequisite for accurate determination of dates and rates across a variety of applications. Increasing the precision of analyses enhances not only the interpretations that can be made from dates and rates, but also enables approaches involving multiple isotopic systems such as burial dating (Granger and Muzikar, 2001) and isochron burial dating (Balco and Rovey, 2008), and allows for improved calibration of cosmogenic nuclide production rates (Balco et al., 2009; Borchers et al., 2016; Briner et al., 2012; Putnam et al., 2010). Very low concentration samples, such as those from young exposures (Licciardi et al., 2009), rapidly eroding landscapes (Portenga et al., 2015), or long-buried sediments (Erlanger et al., 2012; Gibbon et al., 2014), require low detection limits to be measurable above background levels. High $^9\text{Be}^{3+}$ beam currents reduce the counting times required to achieve desired precisions,

thereby speeding AMS throughput and better utilizing the limited beam time available for analysis.

This paper discusses optimization of the Be extraction procedure (Fig. 1) used at the University of Vermont, with the aim of increasing data accuracy and precision as well as the efficiency of sample preparation and AMS analysis. Our goal is to produce pure samples of Be that match the performance of standards, with particular focus on obtaining high Be yield, consistent $^9\text{Be}^{3+}$ beam currents, low and consistent $^{10}\text{Be}/^9\text{Be}$ background levels, and minimal isobaric interference. The optimization strategies discussed here are generalizable to other laboratories as well as to other AMS facilities.

2. Brief history of ^{10}Be extraction and measurement

Over time, different methods have been used to measure terrestrial cosmogenic ^{10}Be . Initially, abundances of cosmogenic ^{10}Be were quantified by radioactive decay counting after Be was isolated from silicate minerals by dissolution in acid (Fairhall, 1960). However, only samples with the highest ^{10}Be concentrations, for example those exposed at high elevations for long durations, could be measured. Later, it became possible to measure $^{10}\text{Be}/^9\text{Be}$ ratios via AMS (Lanford et al., 1980; Raisbeck et al., 1978; Southon et al., 1983; Thomas et al., 1981; Turekian et al., 1979), including on lower-energy AMS systems (Raisbeck et al., 1987). Be yields from chemical preparation were typically high (85–90%), but samples frequently retained impurities, especially Al (Lanford et al., 1980). At that time, precisions were generally 5–10%, and detection was limited to $^{10}\text{Be}/^9\text{Be}$ ratios greater than $\sim 10^{-13}$ (Southon et al., 1983).

Although measurements of ^{10}Be became more common into the 1980's, average AMS beam currents remained relatively low (Klein and Middleton, 1984). By 1990, the quality of AMS measurements of ^{10}Be increased, and precisions of several percent were attainable (Suter, 1990). At around the same time, *in situ* ^{10}Be became a more widely used dating technique as AMS analysis improved and after it was confirmed that meteoric ^{10}Be produced in the atmosphere and adhered to the surface of grains could be removed from the grain coatings of a sample with repeated acid etches (Brown et al., 1991; Kohl and Nishiizumi, 1992; Nishiizumi et al., 1991, 1986, 1989). Adding column chromatography to the extraction protocol ensured that Be could be cleanly separated from other elements (Ditchburn and Whitehead, 1994; Tera et al., 1986).

Recent methodological advances have further increased the quality of AMS ^{10}Be measurements by improving beam currents. BeO had traditionally been mixed with Ag before being packing into cathodes for AMS analysis; however, using Nb instead of Ag increased $^9\text{Be}^{3+}$ beam currents (Hunt et al., 2006; Merchel et al., 2008). It is uncertain whether impurities in the final Be fraction decrease AMS $^9\text{Be}^{3+}$ beam currents beyond dilution effects. Merchel et al. (2008) suggested that additions of Ti did not directly decrease $^9\text{Be}^{3+}$ beam currents, although the resulting dilution of Be did. However, Hunt et al. (2008) found that Al and Ti both depressed $^9\text{Be}^{3+}$ beam currents beyond the effects of dilution (although Ca, Fe, Mg, and Mn did not).

Detection limits have also improved over the past several decades. The discovery that commercial aluminum often contains non-negligible amounts of ^{10}Be occasioned the use of stainless steel and copper cathodes for sample analysis (Middleton et al., 1994), lowering backgrounds. Although commercially-available ^9Be carrier is commonly used, its $^{10}\text{Be}/^9\text{Be}$ ratio is $\sim 10^{-14}$, which hinders the analysis of low-level samples. In contrast, ^9Be carriers made from deeply-mined phenakite (Be_2SiO_4) and beryl ($\text{Be}_3\text{Al}_2\text{Si}_6\text{O}_{18}$) often yield $^{10}\text{Be}/^9\text{Be}$ ratios two orders of magnitude lower (Merchel et al., 2008), which are better suited for the analysis of low-level

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