METEORIC ^{10}Be AS A TRACER FOR SUBGLACIAL CHEMICAL WEATHERING
IN EAST ANTARCTICA

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METEORIC ^{10}Be AS A TRACER FOR SUBGLACIAL CHEMICAL WEATHERING
IN EAST ANTARCTICA

Subglacial chemical processes in Antarctica are potentially significant contributors to global geochemical cycles, but current understanding of their scale and nature is limited. A sequential chemical extraction procedure was developed and tested to investigate the utility of meteoric ^{10}Be as a tracer for chemical weathering processes beneath the East Antarctic Ice Sheet. Subglacial meltwater is widely available under the Antarctic Ice Sheet and chemical constituents within it have the potential to drive geochemical weathering processes in the subglacial environment. Meteoric ^{10}Be is a cosmogenic nuclide with a half-life of 1.39×10^6 years that is incorporated into glacier ice, therefore its abundance in the subglacial environment in Antarctica is meltwater dependent. It is known to adsorb to fine-grained particles in aqueous solution, precipitate with amorphous oxides, and/or be incorporated into authigenic clay structures during chemical weathering. The presence of ^{10}Be in weathering products derived from beneath the ice therefore indicates chemical weathering processes in the subglacial environment. Freshly emerging subglacial sediments from the Mt. Achnar blue ice moraine were subject to chemical extractions where these weathering phases were isolated and ^{10}Be concentrations therein quantified. Optimization of the phase isolation was developed by examining the effects of each extraction on the sample mineralogy and chemical composition. Experiments on ^{10}Be desorption revealed that pH 3.2-3.5 was optimal for the extraction of adsorbed ^{10}Be . Vigorous disaggregation of the samples before grain size separations and acid extractions is crucial due to the preferential fractionation of the nuclide with clay-sized particles. ^{10}Be concentrations of $2\text{--}22 \times 10^7$ atoms g^{-1} measured in oxides and clay minerals in freshly emerging sediments strongly indicate subglacial chemical weathering in the catchment of the Mt. Achnar moraine. Sediment-meltwater contact in the system was calculated to be on the order of thousands of years, based on total ^{10}Be sample concentrations, local basal melt rates, and ^{10}Be ice concentrations. Strong correlation ($R = 0.96$) between ^{10}Be and smectite abundance in the sediments indicate authigenic clay formation in the subglacial environment. This study shows that

meteoric ^{10}Be is a useful tool to characterize subglacial geochemical weathering processes under the Antarctic Ice Sheet.

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1. Introduction

Chemical weathering of Earth materials plays a key role in major global geochemical cycles that regulate the Earth's climate, especially the carbon and sulfur cycles. Antarctica's land area accounts for $\sim 10\%$ of Earth's total land surface but is almost entirely covered by the Antarctic Ice sheet (AIS), the world's largest ice sheet. Subglacial chemical weathering on continental-scales in Antarctica can potentially be a major contributor to the mass balance of geochemical cycles (Graly et al., 2020; Petsch, 2014; Wadham et al., 2012).

Most geochemical studies in Antarctica have focused on chemical solutes in water samples from relatively rare geological features or water outburst events. These include the perennially ice covered freshwater Lake Untersee (Wand et al., 1997), Subglacial Lake Whillans (Michaud et al., 2016), upwelling subglacial brines at Lewis Cliff Ice Tongue (Fitzpatrick et al., 1990), sediment pore waters of Kamb Ice Stream and Bindschadler Ice Stream (Skidmore et al., 2010), water emerging at Blood Falls in the Dry Valleys, and water from a jökulhlaup near Casey Station (Badgeley et al., 2017; Goodwin, 1988; Mikucki et al., 2004). Water samples from these studies may not be representative of the general geochemistry beneath the AIS, but rather characterize water chemistry of those rare or unique geological features within the continent. Model studies have shown that 55% of the grounded ice in Antarctica is at pressure melting point (Pattyn, 2010). Therefore, by areal extent, most of the water on the Antarctic continent is basal meltwater sourced by the geothermal and strain heating under the ice (typically $2\text{-}6\text{ cm yr}^{-1}$) (Pattyn, 2010). This study explores an alternative approach, where subglacially-derived sediments are analyzed as an indicator of subglacial weathering rates across a broader, more integrated portion of the continent.

In the presence of meltwater released from subglacial ice and the atmospheric gases therein, sediments that originate from the comminution of rock at the ice bed can be chemically altered in the subglacial environment (Graly et al., 2020). These sediments become accessible for sampling once they reach the surface of the ice sheet. This is known to occur at glacial moraines scattered across the continent, particularly along the Transantarctic Mountains (Palmer et al., 2012). In order to utilize subglacially-derived sediments as proxies for the subglacial processes, a method that characterizes the

weathering signatures of the sediments needs to be developed which can be applied to locations where subglacially-derived sediments can be collected.

To investigate the possibility of authigenic mineral formation under the ice through chemical weathering, a sequential chemical extraction procedure was modified for use on freshly emerging sediments at the Mt. Achnar Moraine. The technique is widely used to investigate the origins, mode of occurrence, mobility, biological and physiochemical availability of trace elements in Earth materials (Du Laing et al., 2010). Sequential extraction schemes have been developed for a wide range of elements, taking into account the chemical properties and soil behaviors of the targeted elements (Filgueiras et al., 2002). Although the technique has been widely used and adjusted to various elements, the lack of selectivity of the extractants necessitates a careful evaluation of the standard extraction procedures. This is especially true when it comes to elements other than Fe, which has been the most commonly targeted element using the technique (Bacon & Davidson, 2008; Du Laing, 2011).

In this study, we use the cosmogenic nuclide meteoric ^{10}Be as a tracer for subglacial chemical weathering at Mt. Achnar Moraine. The sediments of the moraine bear strong evidence for subglacial chemical weathering at the site, namely abundant weathering products and a decline in magnetic susceptibility compared to the source rock (Grady et al., 2020). Beryllium is known to adsorb to fine-grained soil particles in aqueous solution, to precipitate with oxides and oxyhydroxides, and be incorporated into authigenic clay structures during chemical weathering (Barg et al., 1997; You et al., 1989). These are the main soil behavior characteristics of beryllium used as a framework for a sequential chemical extraction scheme in this study. The aim is to quantify meteoric ^{10}Be concentrations in the various weathering phases and use them to characterize the chemical weathering processes in the catchment area of the moraine.

2. Background and Previous Studies

2.1. Chemical Weathering

Chemical weathering is a process in which Earth materials are chemically and mineralogically altered. This can take place following several different pathways, depending on the environment and the source rock. Chemical weathering typically requires water and dissolved atmospheric gases to produce acids which dissolve primary minerals, e.g. pyroxenes and feldspars. Where chemical weathering occurs, H^+ ions in the acids substitute for cations in the primary mineral lattices, breaking down the structure of the mineral as the cations are released into solution. Secondary minerals can form as a result of these reactions by the alteration of the primary mineral (incongruent reaction) and from precipitation or recrystallization of dissolved constituents in the solution (congruent reaction) (Barton, 2002). The latter is often referred to as the neoformation or authigenic formation of secondary minerals. The type of secondary mineral that forms depends on the chemical composition of the source rock and the solution, the geometric arrangement of the atoms that are available, and the electric forces that bind them together (Klein & Hurlbut, 1985).

Of the eight elements that constitute over 99% of Earth's crust, the O^{2-} anion is by far the most abundant and is a major component in minerals and other Earth materials, e.g. soils. Oxygen is a highly electronegative element that readily forms compounds with other elements. Its binary compound with other elements (oxides), sometimes including the hydroxide ion (OH^- ; oxyhydroxides), forms under conditions of dissolution and neoformation during chemical weathering (Velde, 2013). These amorphous, non-crystalline phases are commonly found in soils, often as grain coatings, and are chemically active compounds which participate in weathering processes like oxidation-reduction and adsorption of cations (Cornell & Schwertmann, 2003; Velde, 2013). In oxidizing environments, amorphous oxides and oxyhydroxides can further evolve to more stable and crystalline structures over time (Du Laing, 2011).

Silica (Si^{4+}) is the second most prevalent element in Earth's crust and forms the SiO_4 tetrahedron with four oxygen ions (O^-), which is the foundational building block of silicate mineral structures (Barton, 2002). SiO_4 tetrahedra can share oxygen ions, in which interlocking arrays of tetrahedra form a tetrahedral sheet. External ions, for

example Al^{3+} , Fe^{2+} and Mg^{2+} , surrounded by six O^- ions, make up an octahedral structure, which can bind with the silicate sheets via one hydroxyl and two oxygen anion groups. Cations in the octahedral structures can either be two or three in number, depending on the valency of the cation. In the case of trivalent cations (e.g. Al^{3+}), two cations are present, making it a dioctahedral structure. When the cations are divalent (e.g. Mg^{2+}), three cations are present and the structure is trioctahedral. The governing factor here is that a positive charge of six is maintained (Velde, 2013). Together, the SiO_4 tetrahedral sheets interlocked with the octahedral sheets make up sheet silicates, or clay minerals (Barton, 2002; Velde, 2013).

Clay minerals are characterized by small sizes ($< 2 \mu\text{m}$ diameter) and large surface areas due to their sheet structures. The charged surfaces of clay minerals attract charged ions in solution due to electrostatic forces that cause them to adsorb to the clay surfaces or between structural sheets. This behavior is termed cation exchange capacity and is the main pathway for clays to affect metal and trace element activity in aqueous solution (Velde, 2013). Isomorphous substitution, the replacement of structural cations within clay structures can occur when atoms with compatible charge-to-radius ratios are present in the solution or on the clay surface (Barton, 2002).

In general, the behavior of trace elements in Earth systems is dependent on their chemical form of occurrence, which is largely governed by redox conditions. Redox conditions affect the availability of species for sorption, precipitation, complexation and crystallization. Typical forms of elements, ordered by increased immobility/complexations are: water-soluble, free ions $>$ exchangeable metals $>$ metals in precipitated inorganic compounds (e.g. carbonates) $>$ metals complexed with humic materials $>$ metals adsorbed to precipitated hydrous oxides $>$ metals bound within crystal structures (Du Laing, 2011). These processes that release and sequester elements in Earth's systems are the drivers of Earth's geochemical cycles.

2.2. Beryllium: Atomic Properties and Crystal Chemistry

Beryllium is an alkaline Earth metal with the atomic number 4 and the ground-state electron configuration $[\text{He}]2s^2$. It most often coordinates as the Be^{2+} cation and predominantly forms covalent bonds due to having only two electrons to contribute to

bond formation and its high electronegativity. Be^{2+} has the smallest radius of all metal ions ($0.27\text{\AA}$) and a high charge-to-radius ratio, making it a strong Lewis acid (electron pair acceptor). As a highly polarizing element, it has a strong affinity for oxygen and thus is capable of orienting water molecules around itself (Everest, 1964; Hawthorne & Huminicki, 2002). Beryllium exists exclusively in tetrahedral coordination in minerals (Hawthorne & Huminicki, 2002). Be^{2+} substitution into octahedral sites causes structural collapse (Wang et al., 2017) and breaking of strong bonds to facilitate tetrahedral coordination of the ion (Buchner, 2020). In minerals, beryllium either exists as an essential cation which cannot be replaced by other cations due to its uniquely small size, or as a substitute cation, commonly for Si and Al, in tetrahedral sites in silicate minerals (Hawthorne & Huminicki, 2002; Vesely et al., 2018). Four oxygen ions surround the Be^{2+} ion in its compounds, forming compact $[\text{BeO}_4]^{6-}$ tetrahedra with strong resemblance to the silica tetrahedron $[\text{SiO}_4]^{4-}$ (Beus, 1956). This, and the similarity to a somewhat lesser extent between the $[\text{BeO}_4]^{6-}$ and $[\text{AlO}_4]^{5-}$ tetrahedra, is an important factor in the geochemical behavior of beryllium (Beus, 1956). Because of their similar ionic radii, beryllium can substitute for the Si^{4+} ion ($0.39\text{\AA}$) within SiO_4 tetrahedra in silicate minerals (Beus, 1956; Rankama & Sahama, 1950). The chemical behavior of Al^{3+} is similar to Be^{2+} due to similar charge densities, electronegativities and charge-to-size ratio of the two ions; beryllium replacement of aluminum in aluminosilicates can occur in the presence of a hydroxide (Beus, 1956). Mn and Mg are also major cations for which beryllium is known to substitute (Everest et al., 1973; Ryan, 2002).

Beryllium is a rare element and is most commonly encountered as a trace metal in low abundances (ppm) in Earth systems (Hawthorne & Huminicki, 2002; Ryan, 2002). As a lithophile element it preferentially partitions into silicate minerals as opposed to sulfides or metals. It has been found to show preference for clay minerals over Fe-oxides once weathered from silicates and available in solution (Beus, 1956; Lum & Gammon, 1985).

2.3. Beryllium Behavior in Soils and Aqueous Solution

^{10}Be has a strong propensity to adhere to fine-grained particles, with a partition coefficient (K_d) on the order of $\sim 10^5 \text{ ml g}^{-1}$ at neutral conditions ($\text{pH} \geq 6$; Fig. 1) (You et al., 1989). It is retained by adsorption to particle surfaces, or by incorporation into

organic matter, clay structures and fine aluminosilicates. During chemical weathering, it also precipitates with Fe to form oxyhydroxides (Barg et al., 1997; Jungers et al., 2009; McHargue & Damon, 1991; Nyffeler et al., 1984).

You et al. (1989) demonstrated the strong dependency of the K_d of beryllium on the pH level in aquatic systems, which can be explained by the speciation of Be in aquatic solutions (Fig. 1). They used multiple materials in river water in their study (kaolinite, illite and river mud), from which they derived a K_d trend increasing with increased pH from pH 0 ($K_d \sim 10^1$) to pH 6 ($K_d \sim 10^{5.5}$). An increase of about four orders of magnitude occurred between pH 2 and pH 6, and a relatively stable constant value from pH 6 to pH 12 ($K_d \sim 10^5$). The hydrolysis products of ^{10}Be are controlled by Be^{2+} , $\text{Be}(\text{OH})_2$ and $\text{Be}(\text{OH})_3^-$ at pH 2-14. The most predominant species at pH < 6 are Be^{2+} and $\text{Be}(\text{OH})^+$. $\text{Be}(\text{OH})_2$ occurs as a solute at around pH 5. It starts precipitating out of solution as a solid around pH 6 up to pH ~12, and is the only species present between pH 8-10 (Fig. 1) (Aldahan et al., 1999; Brown et al., 1992; Takahashi et al., 1998).

Figure 1. The pH dependence of the speciation and distribution coefficient (K_d) of beryllium in aqueous systems, showing the exponential increase in adsorption between pH 3-6. Modified from Brown et al., 1992 and You et al., 1989.

Aldahan et al. (1999) ascribed the constancy of the K_d values between pH 6 and pH 12 to the low solubility of $\text{Be}(\text{OH})_2\text{-solid}$, which forms and precipitates rapidly between pH 6 and pH 10 and thus Be is neither sorbed nor in solution. The precipitation of

Be(OH)₂ occurs regardless of the chemical and physical characteristics of the solution, and suspended particles. Be(OH)_{2-solid} is dominant at pH 6-10, thus K_d values at this range of pH are not valid as Be exists as Be(OH)₂ precipitate, not sorbed or in solution.

Adsorption experiments, with equilibration times from three minutes up to 108 days (Li et al., 1984; Nyffeler et al., 1984; You et al., 1989), have shown that the sorption process takes place in two steps. The first step occurs within the first day, where K_d increases rapidly, and is thought to involve the easily accessible exchange sites of the solids being occupied by Be. K_d increases at a slower rate in the second step, which might indicate that the exchange of Be takes place through slow diffusion toward less accessible sites, possibly inside the crystal lattices (Nyffeler et al., 1984; You et al., 1989). The reversibility of the adsorption process has been demonstrated with desorption experiments and appears to happen in two steps as described for the adsorption process, with a rapid decrease of K_d in the first day and then slowly decreasing K_d values after that (Li et al., 1984; Nyffeler et al., 1984; You et al., 1989). Boschi and Willenbring (2021) found that sorbed ¹⁰Be concentrations correlate with increased cation exchange capacity (CEC) and the inverse percentage of quartz in the system.

Aldahan et al. (1999) studied and compared the sorption behavior of ¹⁰Be between albite (framework silicate structure) and biotite (sheet silicate structure, mica). In accordance with beryllium's high propensity to sorb onto fine particles with high surface area per mass (Shen et al., 2004), they found that, under the same conditions, the amount of beryllium sorbed onto biotite was up to 40 times higher, and the K_d up to 30 times higher, than the values observed for albite. They also observed similar K_d-pH sorption patterns of biotite as You et al. (1989) had demonstrated for kaolinite and illite. Illite has been found to retain beryllium better than montmorillonite and kaolinite in solutions with high ionic strength (You et al., 1989). Boschi and Willenbring (2016) discussed this enhanced retention by illite as the result of its expandable structure. Montmorillonite-group clays also expand and contract in response to the adsorbed cations and have higher CEC than illite, but beryllium is more easily desorbed from montmorillonite (Kabata-Pendias & Pendias, 1984). Boschi and Willenbring (2016) proposed that beryllium's association with montmorillonite involves more reversible, outer-sphere processes (electrostatic interactions of hydrated Be ions with the sorption site ligands), while inner-

sphere processes (involving chemical reaction between Be and the sorption site) and physical inclusions within collapsed interlayers characterize illite's retention of Be. Potassium ions occupy the interlayers of illite, but are lacking on the sheet edges, making the clay susceptible to infiltration by other cations, such as sodium, and expansion during cation exchange. Expansion of the clay structure allows beryllium atoms to enter the interlayers and when the lattice later contracts, the beryllium is locked in, which makes it more resistant to desorption. However, the opposite is true in ultrapure water, where more Be is retained by montmorillonite than illite, which demonstrates the effects of solution ionic strength on illite's retention of Be (Boschi & Willenbring, 2016).

If ^{10}Be is available in the soil where new clay minerals form during rock weathering, it will be incorporated into the authigenic clay lattices (Kabata-Pendias & Pendias, 1984). Barg et al. (1997) found that 42-92% of the ^{10}Be in the lower B and C horizons of the soil profiles in their study was contained within the mineral lattices of authigenic clays.

Beryllium is also incorporated as a cation in oxyhydroxides (Barg et al., 1997; Takahashi et al., 1998), whose quantity in soils is correlated with the percentage of dithionite-citrate extractable Al, and to a lesser degree, Fe (Graly et al., 2010; Jungers et al., 2009). Al and Be show comparable adsorption behavior in soils as their charge-ionic radius ratio is similar, and thus Be is known to replace Al in soil materials (Barg et al., 1997). Singleton et al. (2016) suggested that the retention of beryllium in sediments is mainly controlled by the presence and amount of grain coatings, composed of HCl-extractable Fe, Mg, Ca and Mn phases. The concentrations of these HCl-extractable materials show grain-size dependence, with the highest concentrations found on $<63\ \mu\text{m}$ and 500-850 μm size grains. Furthermore, they found ^{10}Be to be twice as high in the $<63\ \mu\text{m}$ fraction as in the rest of the size fractions. Their data further support ^{10}Be association with iron oxides, both in crystalline and amorphous phases, and ^{10}Be high affinity to fine soil material ($<63\ \mu\text{m}$) (McKean et al., 1993).

2.4. Meteoric ^{10}Be : Production and Deposition

Meteoric ^{10}Be is a cosmogenic nuclide with a half-life of 1.39×10^6 years (Korschinek et al., 2010). It is formed by spallation reactions when galactic cosmic rays (GCR) interact with atoms in the atmosphere (primarily ^{14}N and ^{16}O ; Fig. 2) (Morris et al., 2002). The GCR must penetrate the Earth's geomagnetic field in order to interact with atmospheric materials. However, the geomagnetic field acts as a shield and deflects the rays away from the Earth, unless they exceed a certain cut-off rigidity. This cut-off rigidity varies with latitude as it is determined by the geomagnetic field strength, the

Figure 2. The production and deposition of meteoric ^{10}Be is latitude dependent due to the deflecting forces of Earth's geomagnetic field acting on the galactic cosmic rays (GCR) responsible for the production of the nuclide. The highest GCR flux rates are at the poles, and decrease with decreased latitude, resulting in the lowest flux at the equator. Top insert: Spallation reactions between GCR and N and O in the atmosphere, and the generation of a cascade of secondary cosmic rays, resulting in the production of meteoric ^{10}Be . Bottom insert: ^{10}Be produced in the stratosphere enters the troposphere through the STE in the midlatitudes, and is mainly deposited with precipitation (wet fallout). This causes ^{10}Be deposition to be strongest in the subtropics, where the world's highest soil ^{10}Be concentrations are found, and lower deposition rates at higher latitudes. Middle insert: ^{10}Be deposition at the poles is low due to low precipitation rates, therefore deposition by wet and dry fallout are equally prevalent in Antarctica.

particle's electrical charge, energy, and angle of incidence (Fig. 2). This creates a latitude dependence of GCR flux, with the highest rates at the poles, and the lowest at the equator (Heikkilä et al., 2009; Morris et al., 2002). Cosmic rays that penetrate the geomagnetic field and enter the Earth's atmosphere trigger a reaction (spallation) with atmospheric materials, producing a cascade of secondary cosmic rays. Many of those secondary cosmic rays have enough energy to initiate further collisions, producing more cosmogenic nuclides (Fig. 2) (Masarik & Beer, 1999).

The majority (50-70%) of ^{10}Be is produced in the stratosphere, where it adheres to aerosols, flows with the airmasses for an average of 1-2 years, and mixes throughout the stratosphere (Heikkilä et al., 2008; Masarik & Beer, 1999). ^{10}Be concentrations in the stratosphere mostly represent the latitudinal dependence of production, i.e. lower concentrations at low latitudes and higher concentrations at high altitudes (Heikkilä et al., 2009). Most of the ^{10}Be enters the troposphere in the midlatitudes ($\sim 40^\circ$), where the so-called stratosphere-troposphere exchange (STE) is strongest, between the Hadley and Ferrel circulation cells (Fig. 2) (Heikkilä et al., 2009).

Figure 3. All published ^{10}Be soil concentrations compiled in a global meta-analysis by Graly et al., 2010. The highest concentrations measured are found at low erosion surfaces in well-developed soils in the mid-latitudes.

^{10}Be is mostly removed from the atmosphere at cloud level, where water droplets scavenge the ^{10}Be -bearing aerosols and deliver to Earth's surface. ^{10}Be delivery by precipitation (wet fallout) accounts for 90% of global ^{10}Be deposition (Lal & Peters, 1967). Thus, global deposition rates are heavily dependent on precipitation, with the

highest deposition rates corresponding to the position of the STE in the midlatitudes (Fig. 2) (Field et al., 2006; Heikkilä, 2007). The highest global soil ^{10}Be concentrations are found in well-developed soils with minimal erosion rates in the mid-latitudes due to the leaching of the nuclide down to depth in the soil profile (Fig. 3) (Graly et al., 2010). Dry fallout, where ^{10}Be -bearing aerosols fall to the ground by gravitational pull without being scavenged by precipitation, accounts for only about 10% of the total global ^{10}Be deposition to Earth's surface (Heikkilä et al., 2009). However, the magnitude of dry deposition in the polar regions is comparable to that of wet deposition (Field et al., 2006) and even considered to be the dominant delivery mechanism of ^{10}Be at the poles due to lack of precipitation (Graly et al., 2011).

2.5. Meteoric ^{10}Be in Antarctica

Meteoric ^{10}Be concentrations in Antarctic ice are a function of production, transport and deposition processes, which are affected by the local climate and seasonal changes in stratospheric air influences. Antarctica is located poleward of 60° in the southern hemisphere, where changes in ^{10}Be flux controlled by production are considerably larger than climatically induced changes (Heikkilä et al., 2008). The ^{10}Be flux to Antarctica is modulated by the control of the variations in the geomagnetic field strength and solar activity on the local atmospheric ^{10}Be production rates (Steig et al., 1996) and is considered to be relatively constant at a rate of $\sim 2 \times 10^5$ atoms $\text{cm}^2 \text{yr}^{-1}$ (Graly et al., 2011). Measured concentrations in Holocene ice vary between 2×10^3 and 1.4×10^5 atoms g^{-1} (Aldahan & Possnert, 1998; Horiuchi et al., 2007; Pedro, Heikkilä, et al., 2011; Raisbeck et al., 1981; Raisbeck et al., 1990; Raisbeck et al., 1992; Raisbeck et al., 1987; Steig et al., 1996). In contrast, in-situ ^{10}Be , produced by spallation reactions within crystal lattices exposed on the Earth's surface, is produced at $\sim 3.60\text{--}3.82$ atoms $\text{g}^{-1}_{\text{quartz}} \text{yr}^{-1}$ in Antarctica (Kaplan et al., 2011). In-situ ^{10}Be therefore accumulates far slower than meteoric ^{10}Be .

^{10}Be deposition in Antarctica is heavily regulated by precipitation (Fig. 4). Precipitation is highest on the coast, with a maximum in areas closest to South America and west of it, although the differences are small. Precipitation rates decrease inland, reaching minimum in central East Antarctica. In the driest areas, dry deposition can reach

up to 50% of the total ^{10}Be deposition (Heikkilä, 2007). Concentrations of ^{10}Be in the Antarctic ice sheet are therefore dependent on snow accumulation, with higher concentrations accumulating with low snowfall, and lower concentrations when snowfall is higher. This is evident in ice core records from Dome C and Vostok covering the last 30 ky, where ^{10}Be concentrations from the late Pleistocene are higher than concentrations from the Holocene by a factor of 2-3 (Raisbeck et al., 1981; 1987). The difference in concentrations between the Holocene and the last ice age shows the climatological effect on meteoric ^{10}Be concentrations in ice. ^{10}Be flux was relatively constant during those periods, but higher ice concentrations accumulated during colder periods when snowfall was limited (Raisbeck et al., 1981; Raisbeck et al., 1987). Higher precipitation rates in the Holocene caused a diluting effect, resulting in lower ^{10}Be concentrations in the ice (Raisbeck et al., 1987; Yiou et al., 1985). The Mt. Achernar Moraine ice is sourced from the polar plateau and has stable isotopes signatures that indicate formation during the Pleistocene (Graly et al., 2018b). Ice core records from Vostok, which is the closest core to Mt. Achernar where ^{10}Be has been measured, show meteoric ^{10}Be concentrations of $\sim 2 \times 10^5$ atoms g^{-1} (Raisbeck et al., 1990). For the purposes of this study, meteoric ^{10}Be concentrations of $\sim 2 \times 10^5$ atoms g^{-1} are assumed in the Mt. Achernar moraine system.

Figure 4. Relative ^{10}Be concentrations in the Antarctic Ice Sheet (a) compared to (b) precipitation rates in Antarctica (mm day^{-1}). Vostok is the core closest to Mt. Achernar. Aldahan et al. (1998); Horiuchi et al. (2007); Pedro, Smith, et al. (2011); Raisbeck et al. (1981); Raisbeck et al. (1990); Steig et al. (1996)st (dark blue). Ice core and snow pit data from Aldahan et al. (1998); Horiuchi et al. (2007); Pedro, Smith, et al. (2011); Raisbeck et al. (1981); Raisbeck et al. (1990); Steig et al. (1996). (b). modified from Heikkilä (2007).

2.6. ^{10}Be as a Tracer for Subglacial Chemical Weathering: Phase Isolation using Sequential Chemical Extractions

Studies and development of the use of meteoric ^{10}Be as a tracer for soil and sediment processes have largely been focused on temperate environments but is increasingly gaining momentum in the field of glacial studies (Graly et al., 2018; Schiller et al., 2009; White et al., 2019). Antarctica has been glaciated for over 3×10^7 years (DeConto & Pollard, 2003; Pollard & DeConto, 2009), or three times the applicability of the meteoric ^{10}Be signal. Any meteoric ^{10}Be deposited on the continent prior to glaciation is therefore depleted. Most of the ^{10}Be that is currently found on the continent (deposited in the past 10^7 years) has settled on and accumulated in the ice sheet. This study proposes a sequential chemical extraction procedure which takes advantage of the isotopic and chemical properties of meteoric ^{10}Be .

Three main sediment fractions in which Be is known to exist: adsorbed in aqueous systems, precipitated with amorphous oxides and oxyhydroxides and incorporated into authigenic clay lattices (Barg et al., 1997; Wittmann et al., 2012; You et al., 1989), are proposed as indicators for the degree of subglacial chemical weathering. Highly weathered, freshly exposed samples of subglacial origin at the Mt. Achnar moraine (Graly et al., 2020) were subject to sequential chemical extractions designed to isolate the three defined phases of ^{10}Be . A detectable ^{10}Be signal in these sediments would indicate the accumulation of the nuclide into those sediments from the ice due to their relatively short surface exposure of $< 10 \times 10^3$ years (Graly et al., 2018a). For ^{10}Be to adsorb to the surface of the sediment particles, the beryllium would have to be released from the ice as it melted and come into contact with the sediment through the glacial melt water. Meteoric ^{10}Be measured in amorphous phases such as oxides or oxyhydroxides would indicate the precipitation of those species in the subglacial environment. Lastly, if ^{10}Be is measured in clay structures, that would indicate the authigenic formation of clays in the subglacial environment. Due to the $> 3 \times 10^7$ -year glaciation history of the area (DeConto & Pollard, 2003; Pollard & DeConto, 2009), inheritance of meteoric ^{10}Be from the source rock is unlikely. The applicability of the nuclide is $< 10 \times 10^6$ years is due to the 1.39×10^6 year half-life of meteoric ^{10}Be (Korschinek et al., 2010). That is, all meteoric ^{10}Be which was deposited onto the bedrock prior to glaciation has been fully

depleted for over 2×10^7 years. The relative fractionation of ^{10}Be between the three phases could elucidate the chemical weathering activity under the ice, i.e., the higher degree of complexation of ^{10}Be , the higher the degree of weathering. (Fig. 5).

Figure 5. The conceptual model of the study: atmospherically deposited meteoric ^{10}Be being delivered from the ice surface, to the subglacial environment via ice flux and finally emerging at the surface at a blue ice moraine. The fractionation scheme of meteoric ^{10}Be in the sediments originated in the catchment of Law Glacier and collected at the active margin of the Mt. Achnar Moraine. Ice cover has prevailed for $> 3 \times 10^7$ years, meaning that any detectable meteoric ^{10}Be concentrations in the freshly emerging sediments at the moraine must have accumulated via chemical processes in the subglacial environment after being released from the ice into meltwater. Adsorbed ^{10}Be would therefore indicate the presence of meltwater, ^{10}Be in oxides or oxyhydroxides would indicate the precipitation of those phases, and ^{10}Be in clay structures would indicate the authigenic formation of clays in the subglacial environment. Subglacial sediments become entrained in the basal ice layers and emerge on the surface in blue ice areas due to high sublimation rates and upward flow trajectories of the ice.

Sequential chemical extractions are a technique where solid samples are subject to a series of progressively stronger acids, each designed to selectively dissolve specific components of the sediment particles (Berner et al., 1977; Tessier et al., 1979; Wiederhold et al., 2007). Sequential extraction schemes typically involve the chemical separation of trace elements into: 1) fractions soluble in water, 2) exchangeable and/or acid soluble fractions (trace elements associated with carbonates), 3) reducible (mainly Fe and Mn oxides), 4) oxidizable (metals bound to organic matter), and 5) residual/silicate fractions (crystalline-bound trace elements) (Du Laing, 2011). Tessier et al. (1979) proposed a procedure which has been widely used and developed for a wide range of elements, where the specific chemical properties of the target element have been taken into account (Du Laing, 2011). Although widely used in Earth science research, the technique suffers from the lack of selectivity of the extractants. It is an operationally defined scheme and assessments of whether the common procedures accurately dissolve the targeted sample fractions are lacking (Slotznick et al., 2020). In this study, we seek to isolate three sediment fractions in which meteoric ^{10}Be is likely found: 1) adsorbed, 2) reducible oxides and oxyhydroxides, and 3) residual clay and silicate fractions, in order to assess the chemical weathering activity in the subglacial environment at the Mt. Achernar Moraine. The sequential extraction procedure was carefully examined and developed to optimize the isolation of the defined ^{10}Be phases.

2.7. Study Area

2.7.1. The Transantarctic Mountains

East and West Antarctica are divided by the 100-200 km wide Transantarctic Mountains (TAM), which extend 3500 km across the continent and reach > 4500 m elevation (Fitzgerald, 2002). The history of the TAM is revealed by exposed bedrock in mountain ranges and individual mountain peaks (nunataks), representing events of magmatism, metamorphism, exhumation, sedimentation from East and West Antarctica. The basement of the central TAM consists of deformed and metamorphosed Neoproterozoic-Cambrian sedimentary and volcanic rocks of the Beardmore and Ross Orogens, and granitoid batholith intrusions of the Granite Harbour Intrusives (Elliot, 1975; Fitzgerald, 2002). Next in the sequence near Mt. Achernar are Permo-Triassic

strata, composed of glacial, alluvial and shallow marine sediments. This stratigraphic sequence is part of the Beacon Supergroup, which is exposed at Mt. Achnar (Barrett et al., 1986). The Beacon Supergroup is intruded by mafic sills of the expansive Ferrar Dolerite at Mt. Achnar (Isbell, 1990). The sediments analyzed in this study are sourced from the local bedrock formations (Bader et al., 2017) and thus their properties are an important factor when inferring the subglacial processes that take place at Mt. Achnar.

The upper part of the Beacon Supergroup is the Permian-Triassic Victoria Group, terrestrial sediments derived from East Antarctica and volcanoclastic, arc-derived sands from West Antarctica (Elliot et al., 2017). Massive, stratified diamictites and poorly sorted sandstones of the glaciogenic Pagoda tillite are the oldest formation of the Victoria Group (Elliot et al., 2017). The Mackellar, Fairchild, and Buckley Formations comprise the remaining lower Victoria Group and are associated with deposition along the Gondwana margin. These formations mostly consist of alluvial sediments derived from East Antarctica. Arc-derived volcanoclastic material in the upper Buckley Formation marks a shift in sources from the East Antarctic craton to West Antarctic magmatic arc (Elliot et al., 2017). The rest of the Victoria Group: the Upper Buckley, Fremouw, and Falla Formations consist of shifting East and West Antarctic sources, indicating changes in paleo currents (Barrett, 1991; Elliot et al., 2015; Elliot et al., 2017). Most formations are not a single rock type and contain interbedded sandstones and coal/carbonaceous shale (Elliot et al., 2017). Organic matter in rock samples from the Victoria Group has been measured at avg. 0.55 %wt. in the Mackellar Formation and avg. 23 wt.% in the Buckley Formation, while the coal seams of the Lower Buckley Formation has > 50 wt.% organic matter (Horner, 1992). Mafic dykes and sills of the Ferrar Dolerite pervasively intrude the Beacon Supergroup. These intrusions are products of a short-lived (<0.4 Ma) period of tholeiitic magmatism initiated at the time of the breakup of Gondwana in the early Jurassic (Elliot, 1975). Local contact metamorphism caused by the Ferrar intrusions and burial diagenesis resulted in the formation of illite, chlorite and zeolite cements (Bernet & Gaupp, 2005).

2.7.2. Blue Ice Areas in the Transantarctic Mountains

The TAM stand as a topographic threshold between the EAIS and West Antarctica. Ice flow from the EAIS flows towards the Ross Embayment and is focused as fast-flowing outlet glaciers through topographic gaps in the TAM. Such conditions favor the formation of blue ice areas, which are characterized by a negative surface mass balance due to high sublimation rates (Bintanja, 1999). Along uneven topography around nunataks, extremely cold and dry descending winds (katabatic winds) erode snow cover and cause high sublimation rates at the ice surface. This lowers the surface of the ice and leaves glacier ice exposed, at the same time as it causes upward flow trajectories of ice towards the surface (Bintanja, 1999). This negative surface mass balance creates a feedback mechanism that sustains the blue ice areas: the lowering of the ice enhances the katabatic winds, which increases sublimation rates that further lower the ice surface (Bintanja, 1999).

Sediment accumulation commonly occurs at the margin of blue ice areas as subglacial sediment emerges with the ascending ice (Fig. 5). Such sediment accumulation areas are called blue ice moraines. They range from being isolated patches of sediment to being up to 100 km² in size (Kassab et al., 2019), and can form over long periods of time (>100 ka) (Altmaier et al., 2010; Kaplan et al., 2017). Debris could be englacial or in basal ice layers and be transported with ice flow. The upward flow trajectories of ice at blue ice areas cause the eventual emergence of entrained sediments at the ice surface (Bintanja, 1999; Whillans & Cassidy, 1983). Blue ice moraines are therefore important repositories of material representative of the otherwise ice-covered subglacial environment (Bader et al., 2017; Graly et al., 2020; Kassab et al., 2019). Rockfall from the surrounding topography can also contribute to sediment accumulation at blue ice moraines. However, the material at the study site bears strong evidence of subglacial origin rather than rockfall (Bader et al., 2017; Graly et al., 2020; Kassab et al., 2019).

2.7.3. Law Glacier and Mt. Achnar Moraine

Law Glacier flows from the EAIS between the Queen Alexandra and Queen Elizabeth Ranges in the central TAM, and feeds into the Bowden Nv. Mt. Achnar is the first exposed nunatak that Law Glacier encounters as it flows from the polar plateau towards the Ross Embayment (Fig. 6A). The regional subglacial topography is high

elevation (Jamieson et al., 2010; Paxman et al., 2019), where modeling studies have found the initial growth of the AIS to have taken place (DeConto & Pollard, 2003; Pollard & DeConto, 2009; Wilson et al., 2012). Potential for pre and interglacial exposure, inheritance and weathering at the site is therefore low.

Permian sandstones and coal seams of the Buckley Formation (Beacon Supergroup) with three Ferrar dolerite sills comprise the bedrock exposed at Mt. Achnar (Barrett et al., 1973). Law Glacier encounters Mt. Achnar as a barrier in its path, where a blue ice area has formed at an elevation of ~1700-1900 m above sea level (asl.). Together, the flow of Law Glacier along Mt. Achnar and sublimation at the blue ice area cause the upward ice-flow trajectories of Law Glacier along the nunatak and the eventual exposure of subglacially-derived sediments at the Mt. Achnar moraine (Fig.

Figure 6. The study area in the Central Transantarctic Mountains, where the East Antarctic Ice Sheet funnels through the Queen Alexandra and Queen Elizabeth Ranges as Law Glacier, resulting a blue ice area where sediment accumulation downstream of Mt. Achnar, known as the Mt. Achnar Moraine, is located. A. Insert in the lower left corner indicating the location on a continental scale (modified from (Howat et al., 2019). Mt. Achnar is marked with a yellow star, and the inferred ~4000 km² catchment area of the Mt. Achnar Moraine sediments from Graly et al., 2020 is shaded in red. B. Sampling locations at the Mt. Achnar Moraine marked as magenta dots. GPS data from Kassab et al., 2019 is included as black arrows which indicate the flow direction and relative magnitude of the ice flow velocity, which is fastest at $\square$ 25 m y⁻¹. White rectangle represents the area of panel C. C. Sample 4Q was collected ~ 2 km inward of the active margin of the moraine, where the rest of the samples were collected. Sample 0B was collected from a debris band outside of the moraine margin. Imagery ©2014 Digital Globe, Inc. provided by the Polar Geospatial Center (St. Paul, Minnesota, USA).

6B) (Bader et al., 2017; Kassab et al., 2019). Modeled warm basal conditions (Pattyn, 2010) and stable isotope signatures of the ice in the catchment area suggest that entrainment of the subglacially derived material at the moraine margin happens by regelation, a warm-based process, approximately 30-50 km upstream (Fig. 6A) (Graly et al., 2018). However, a debris band outside of the moraine shows less evidence for regelation, thus cold-based freeze-on processes might, at least partially, be responsible for the entrainment of those sediments (Graly et al., 2018).

Provenance and detrital zircon analyses on the moraine found that the Mt. Achnar Moraine sediment is derived relatively locally from the bedrock underlying Law Glacier (Bader et al., 2017). The main sources are the Mackellar, Fairchild and Buckley Formations of the Beacon Supergroup, as well as Ferrar dolerite (Bader et al., 2017). Lithologic changes across the moraine represent changes in debris source as Law Glacier has tapped into successively lower subglacial stratigraphic units of the Beacon Supergroup, thereby exposing the stratigraphy underlying Law Glacier in the form of emerging detrital sediments at the Mt. Achnar moraine (Bader et al., 2017). A ground-penetrating-radar (GPR) study of the moraine's subsurface interpreted steep dipping patterns within the moraine to be alternating layers of clean ice and debris bands resulting from the lateral accretion of basal sediment (Bader et al., 2017). Based on surface exposure ages (Kaplan et al., 2017) and boron concentrations (Graly et al., 2018a) lateral debris accumulation rates are 2 to $> 40 \text{ m ka}^{-1}$ and vary over time, with higher accumulation rates occurring in colder climate conditions (Kassab et al., 2019). Approximate time since the entrainment of the sediments $\sim 30\text{-}50 \text{ km}$ upstream is estimated to be $9.5\text{-}13.5 \times 10^4$ years and the emergence time from the glacial bed to the moraine surface approximately 3.5×10^4 years (Graly et al., 2018b; Kassab et al., 2019) (Fig. 6A). The area has been ice-covered for $> 30 \times 10^6$ years (DeConto & Pollard, 2003; Pollard & DeConto, 2009), therefore inheritance of ^{10}Be from the source rock is unlikely.

Mass balance calculations of the $< 63 \text{ }\mu\text{m}$ size fraction showed that 28% of the input to the site is from the Ferrar dolerite and 72% from sand- and siltstones of the Fairchild and Mackellar Formations (Graly et al., 2020). The mineralogical and chemical composition of the subglacially derived $< 63 \text{ }\mu\text{m}$ fraction of sediment freshly emerging at the moraine margin indicate significantly more weathering products than the

representative source rock (Graly et al., 2020). Significant mineralogical changes between the source rock and the weathering products manifest as losses in feldspar and pyroxene, and enrichment in smectite and kaolinite clays, calcite, zeolites and amorphous materials like oxides and oxyhydroxides (Graly et al., 2020). Increase in smectite is 15-fold and 7-fold for kaolinite, suggesting authigenic formation of smectite and kaolinite from weathered feldspars and pyroxenes. X-ray diffraction (XRD) patterns of the clay fractions in the sediments showed broader smectite peaks than those in the rock samples, possibly due to greater disorder in layer stacking and/or several smectite compositions present in the sediments. Magnetic susceptibility measurements found significant magnetite depletion in the sediments relative to the source rock, suggesting the oxidation of magnetite from the source rock (Graly et al., 2020). Carbonation weathering, driven by the oxidation of organic matter from the source rock and oxygen supplied from the melting ice, is likely responsible for the chemical alteration of the source rock at the Mt. Achnar moraine (Graly et al., 2020).

3. Methods

3.1. Sampling

Glacial sediment samples were collected from the Mt. Achernar blue ice moraine during the 2010 and 2015 field seasons. Freshly emerging sediments were either collected directly from ablating ice or from the first crests on the active margin of the moraine. Samples exposed on the moraine surface for up to 5×10^5 years were collected along a 6.5 km transect across the moraine surface (Bader et al., 2017; Kaplan et al., 2017). Samples were collected using an inert plastic spoon and scooped into an inert plastic bag (Fig. 7). Subglacial origins of the samples are suggested by features such as striated or faceted clasts, fine grain sizes, and the presence of various exotic cobbles different in composition from the local bedrock (Bader et al., 2017). Five samples of the freshly emerging sediments and one sample with a 5×10^5 year exposure history (Kaplan et al., 2017), collected about 2 km inward of the moraine margin, were included in this study (Fig. 6C).

Figure 7. Freshly emerging sediment samples collected from the active margin of the moraine still embedded in ice at the time of acquisition.

3.2. Laboratory Procedures

The following methods were the baseline procedures used for all samples, which was adjusted based on results after each analysis in order to optimize the phase isolation. Every sample was treated in the following manner unless otherwise stated in the sections specific to each round of analysis. Step-by-step procedures can be found in appendix B. ^{10}Be concentration measurements were performed by Accelerator Mass Spectrometry (AMS) at the Purdue Rare Isotope Measurement Laboratory (PRIME Lab).

3.2.1. Initial Sample Preparation

All samples were stored in plastic bags after their acquisition during the 2015-16 field season. Dry samples were stored at room temperature and wet samples in a refrigerator at 4°C. Sub-samples used for analysis were removed from the bags with a clean spoon after homogenizing the sample inside the bag. Moisture was removed from wet samples by freeze drying. Dry samples were sieved through a 2 mm mesh sieve to separate gravel-sized grains from smaller grains. Gravel-sized particles were hand-picked from wet samples using clean tweezers.

Grain size dependency was investigated by separating each sample into different grain sizes using wet sieving and gravity-settling based on standard procedures according to Stoke's Law (Chipera et al., 1993; Tanner & Jackson, 1947). See step-by-step procedures in appendix B. The terms used to describe the grain size fractions hereafter are operationally defined and therefore might not follow the Wentworth grain size classification for grains $<63\ \mu\text{m}$. This is due to the uncertainty of the grain sizes which gravity settling yields. After the initial 2 mm dry sieving, the samples were sieved with a 500 μm sieve. They were then suspended in water in glass beakers for 10 minutes. The settled material was wet sieved with a 250 μm sieve, yielding the size fraction termed medium sand in this study, and a 63 μm sieve, yielding the size fraction termed fine sand hereafter. Grains still in suspension after the 10-minute settling were decanted with the water for a subsequent 1-hour settling. Settled material from the 1-hour settling is referred to as silt in this study. Suspended material after the 1-hour settling was decanted and settled for another 15 hours. Materials settled during the 15 hours are referred to as coarse clay, and what was still in suspension is referred to as fine clay hereafter. Medium

sand, fine sand and silt fractions were centrifuged, and the water decanted. The water from the coarse and fine clay fractions was evaporated by leaving the samples in an oven at 62°C until only the moist clays were left. The remaining moisture in all samples was removed by freeze drying.

3.2.2. Experimental Development: Sequential Chemical Extractions

The three defined sediment phases need to be isolated as accurately and completely as possible in order to utilize ^{10}Be concentrations therein as a proxy for subglacial chemical weathering. Acetic acid (CH_3COOH) in 0.11 M concentration was used to target adsorbed species and carbonate content. The dry samples were placed in plastic tubes and 40 ml of the 0.11 M acetic acid added. After shaking the samples completely into suspension, the tubes were placed on a vibrating table for 16 hours. The samples were centrifuged at 3400 x g relative centrifugal force (RCF) for 15 minutes, and the acid and the dissolved components therein (the leachate) was then decanted into new tubes and stored for future analyses. Thirty ml of deionized water were then added to the samples, which were shaken completely into suspension and left on a vibrating table for 2-hours to rinse, then centrifuged at 3400 x g RCF for 15 minutes and the water decanted. This was followed by a 24-hour leach with 0.5 M hydrochloric acid (HCl), which was used to dissolve amorphous/non-crystalline phases such as oxides and oxyhydroxides (Wittmann et al., 2012), following the same procedures as for the acetic acid extraction. What remained of the samples after the acetic and hydrochloric acid extractions was totally dissolved in various mixtures of HCl, nitric acid (HNO_3) and hydrofluoric acid (HF) using microwave digesters. Further details on the total dissolution procedures can be found in sections 3.2.2.3 and 3.2.2.4., and in appendix B. Chemical and mineralogical effects of the sequential chemical extraction procedures were examined using ICP-OES (Inductively Coupled Plasma – Optical Emission Spectrometry) and XRD analyses to assess and optimize the efficacy of the phase isolation. Sample leachates from each extraction were analyzed and the results used to determine whether adjustments were necessary. Comparability was achieved by subjecting a single control sample, 0B, to all experiments. For the final analysis, where total ^{10}Be concentrations and relative phase fractionations were used to infer chemical processes, differences in ^{10}Be yield between the methods was accounted for to allow for a more accurate analysis. This was done by

calculating the differences in ^{10}Be yields of sample 0B between the methods, which was then used to normalize ^{10}Be yields of samples which did not receive the optimal treatment for ^{10}Be extraction.

3.2.2.1. pH Dependency on Element Adsorption and Desorption

To examine the effects of pH on the adsorbed species and oxide and oxyhydroxide grain coatings, all samples were subject to a 16-hour extraction with CH_3COOH (acetic acid) at four different concentrations: 0.05 M, 0.11 M, 0.22 M and 0.44. The pH of each solution was measured before adding 1 g of sample in the $< 63 \mu\text{m}$ size fraction, immediately after sample addition, and every two hours for the remainder of the extraction, using an Oakton 700 pH meter. All other aspects of the procedure were the same as with other acetic acid leaches. Chemical data achieved by ICP-OES analysis was used to assess which species dissolved in each solution and determine the ideal pH levels to target adsorbed species.

3.2.2.2. High pH Extractions using Sodium Carbonate (Na_2CO_3)

Due to the excess of Si from chemical weathering of feldspars and pyroxenes at the study site (Graly et al., 2020), a high-pH extraction was performed to investigate whether amorphous silica phases host significant amounts of ^{10}Be . Three samples were subject to a five-hour $\text{pH} > 11$ extraction in a 2 M sodium carbonate (Na_2CO_3) solution (Sauer et al., 2006). The solution was prepared from Na_2CO_3 powder and Milli-Q water in a glass beaker on a hot plate and the pH measured before combining with the samples. One gram of $< 63 \mu\text{m}$ fraction of each sample the was mixed with 40 ml of the Na_2CO_3 solution in a plastic tube and placed in a sonicating tub at 60°C for five hours (Sauer et al., 2006). Every 15 minutes, the samples were shaken to ensure that the grains remained in suspension and the temperature of the water inside the tub was monitored. After the extraction, the samples were centrifuged at $3400 \times g$ RCF and the extractants decanted.

ICP-OES analysis on the extractants was performed following the same procedures as with all other samples. However, to prevent chemical rebound between samples, the standard 2% HNO_3 flush was switched out for a $\text{pH} > 10$ flush made from sodium carbonate and Milli-Q water while analyzing the extracts.

3.2.2.3. Effects of 0.5 M HCl on Clay Minerals

To determine the effects of 0.5 M HCl on clays, two ~1 g aliquots in the < 63 μm size fraction of samples 0B, 0E and 1B were subject to two sequential extraction methods (Fig. 8). Both methods were the same in all regards except for the timing of clay (<1 μm particles) separations by gravity settling. All samples were leached in 0.11 M acetic acid for 16 hours and water rinsed for two hours, then leached in 0.5 M HCl for 24 hours. Clays were separated after the HCl extraction in method 1 and before the HCl extraction in method 2. All samples were rinsed after the HCl leach, oven dried at 60°C and weighed once they reached room temperature. A mixture of HF, HNO₃ and HCl in the ratio 1:2:2 was used in the total dissolution process. The samples were shaken vigorously to ensure complete particle suspension, sonicated at 50°C for three hours and then digested in a CEM corporation Microwave Digestion System.

Figure 8. Methods 1 and 2: Samples 0B, 0E and 1B were subject to two extraction sequences utilizing the same solutions (0.11 M acetic acid and 0.5 M HCl) and digestion procedures, but with clay separations at different stages in the sequence. In method 1, the clays were separated after the HCl extraction and in method 2, the clays were separated before the HCl extraction. Rectangles with rounded edges represent the sediment phases in which ¹⁰Be was measured.

3.2.2.4. Grain Size Dependency of ^{10}Be Fractionation

To study the distribution of ^{10}Be between different grain sizes, the samples were separated into five different grain sizes and disaggregation of fine particles was incorporated into the procedure. A ~5 g subsample of four samples was weighed and disaggregated by mixing in a Waring Blade BB340SK bar blender for 60 seconds and sonicated using a sonication probe from a Malvern Hydro MU unit for 10 minutes twice before grain size separation (see section 3.2.1. and appendix B step-by-step procedures) (Chipera et al., 1993). The five resulting size fractions; medium sand (MS, 250-500 μm), fine sand (FS, 63-250 μm), silt (St, 2-63 μm), coarse clay (CC) and fine clay (FC), of every sample were dried, weighed and placed in separate plastic tubes. All size fractions were leached for 16 hours with a 0.11 M acetic acid, and a 24-hour HCl extraction was performed on the MS, FS and St size fractions. All sample fractions were totally digested in pertinent mixtures according to the CEM MARS 6 Microwave Digestion manual (CEM, 2018). Medium sand, fine sand and silt were mixed with 5 ml H_2O , 8 ml HF and 3 ml HNO_3 while the coarse and fine clay fractions were digested in 3 ml HNO_3 , 5 ml HF and 1 ml HCl (CEM, 2018) (Fig. 9). Column chromatography was performed at the Tulane University Cosmogenic Nuclide Laboratory (TUCL) following standard procedures (Ditchburn & Whitehead, 1994).

3.2.3. Chemical Analysis: ICP-OES

All extracts and blanks were analyzed in triplicates for major elements on a Perkin Elmer Optima 7000 inductively coupled plasma – optical emission spectrometer (ICP-OES) (step-by-step procedures in appendix B). Due to the difference in pH, the standard 2% HNO_3 flush was switched out for a pH > 10 flush made from sodium carbonate and Milli-Q water while analyzing the extracts from the high pH sodium carbonate extraction. This was done to prevent chemical rebound between samples.

3.2.4. Mineralogical Analysis: XRD

Mineral abundances and clay mineralogy were measured with a Bruker D8 Discover X-ray diffractometer. Powder X-ray diffraction and Rietveld refinement were used to analyze the mineral abundances. The samples were prepared by mixing the < 63 μm size fraction with ~ 20 wt.% corundum standard and crushed in an agate mill to pulverize and homogenize before placing in the sample holder. Measurements were made from 2° to 70° 2θ with a Lynx Eye 1D detector. The patterns were processed by Rietveld refinement (Bish & Post, 1989; Rietveld, 1969) using the software Topas (Bruker AXS, version 5) to quantify mineral abundances. All samples except for 0A and 0E were done previously for another study (Graly et al., 2020). Clay mineralogy samples were prepared by suspending the clay fraction (derived from 1-15 hour Stoke's settling) of the samples in water and pipetting on to a zero-background slide where they would air-dry. After analysis, the samples were placed in a glycol chamber for 48 hours and analyzed again to study the expandable clays within the samples. Clay mineralogy was analyzed from 2° to 30° 2θ . To investigate the effects of acetic acid and HCl on the clay minerals, the clay mineralogy was analyzed before any acid extractions, after the acetic acid extraction and after the subsequent HCl extraction.

3.2.5. Preparation for AMS Isotope Analysis

All extracts and blanks were spiked with 250 μm of a ^9Be standard (1041 ± 8 ppm) prepared at PRIME Lab, Purdue University (Batch number: 2014.10.20-Be). The weight of the sample and spike were recorded with a Mettler Toledo XS250DU weighing scale with 5 decimal points and repeatability of 0.05 mg.

Beryllium was separated from the sample extracts by anion and cation exchange column chromatography at PRIME Lab following standard procedures (Ditchburn & Whitehead, 1994; Ochs & Ivy-Ochs, 1997).

Figure 9. Method 3: Samples 0A, 0B and 1A were subject to sequential extractions after being separated into five different size fractions to investigate the effects of grain size on ^{10}Be fractionation. Acetic acid and HCl at the same concentrations as in methods 1 and 2. Total dissolution solutions differed from methods 1 and 2: 5 ml H_2O , 8 ml HF and 3 ml HNO_3 and both clay fractions were dissolved in 3 ml HNO_3 , 5 ml HF and 1 ml HCl. Rectangles with rounded corners represent the sediment phases in which ^{10}Be was measured.

4. Results

4.1. Sample Mineralogy

The mineralogy of the freshly emerging samples in the <63 μm fraction was analyzed using XRD and Rietveld refinement. The mineralogy of the samples was relatively similar to each other and was primarily dominated by clays, quartz and amorphous material (Table 1). Clay minerals made up 27-38% of the samples, with smectite and illite as the most abundant clay species. Alkali feldspars were more abundant than plagioclase feldspars, at 10-16% and 2-4% respectively. Both orthopyroxene and clinopyroxene content accounted for 1-4% of the sample weight. Calcite and dolomite were detected in trace amounts in some samples, except for sample 1A which contained 2.9% calcite. Chlorite content was below 1% in all samples except for 1A, with 3.6% chlorite by weight. Amorphous content and trace phases below XRD detection were 21-33% of the sample weight.

Sample 4Q, with a 5×10^5 -year exposure history, had similar mineralogical character as the freshly emerging samples. The main difference was that it had significantly higher amorphous content, 43.5% compared to an average of 27.3% in the other samples. Its lower quantities of smectite, and detectable levels of calcite and dolomite were also notable.

Table 2. Results in weight % of Rietveld quantitative XRD analysis of the < 63 μm fraction of the samples.

	Qz.	Pig.	Opyx.	Aug.	Plag.	Orth.	Alb.	Ill.	Chlo.	Kaol.	Smec.	Lam.	Clz.	Cal.	Dol.	Amo.
0A	18.8	1.4	2.0	3.0	2.1	6.5	4.5	14.3	0.3	0.3	12.9	0.8	0.4	-	-	32.8
0B	19.2	3.0	-	3.0	1.8	4.6	7.6	14.7	0.8	2.4	20.0	0.2	-	-	-	22.7
0E	17.2	0.3	1.0	4.4	3.6	5.6	4.4	13.9	0.2	0.5	15.0	1.1	1.6	-	-	31.2
1A	18.3	1.2	1.4	2.6	4.0	4.2	10.4	14.7	3.6	2.2	10.2	1.9	0.8	2.9	-	21.6
1B	21.3	0.6	3.8	0.5	2.1	3.7	12.3	16.8	0.4	0.6	8.7	0.7	0.3	0.3	-	27.9
4Q	11.8	1.0	0.1	1.0	6.1	2.7	7.7	14.7	0.6	1.4	6.7	0.5	-	2.1	0.1	43.5

Qz. – quartz, Pig. – pigeonite, Opyx. – orthopyroxene, Aug. – augite, Plag. – plagioclase, Orth. – orthoclase, Alb. – albite, Ill. – illite, Chlo. – chlorite, Kaol. – kaolinite, Smec. – smectite, Lam. – lamontite, Clz. – clinozoisite, Cal. – calcite, Dol. – dolomite, Amo. – amorphous material.

4.2. Experimental Development

4.2.1. pH Dependency of Element Adsorption/Desorption

Acetic acid extractions at four different concentrations were conducted to assess the pH range at which adsorbed species, including ^{10}Be , desorb without significantly affecting other grain components. Beryllium's adsorption behavior in aqueous solution is highly pH dependent, with exponential increase between pH 2 and 4 (You et al., 1989). Initial pH levels of all concentrations were below 3 immediately after preparing the solutions. The higher the solution concentration, the lower the pH throughout the 16-hour period (Fig. 10A). Sample addition to the solution immediately raised the pH by 0.3-0.9 in all cases (Fig. 10C). Increase in pH continued for four hours, then stabilized with an average change of ± 0.1 between each measurement for the remaining 12 hours (Fig. 10B). All sample pH measurements were in the range of pH 2.90 – 4.25 (Fig. 10B and 10D). All samples were measured at a small range of pH within each solution concentration except for the relatively calcite-rich samples, 1A and 4Q, which were measured at significantly higher pH levels.

Higher quantities of all elements, including meteoric ^{10}Be , were leached from all samples as pH decreased (Fig. 11). The most abundant element was Ca at 0.27-2.00% of the sample weight, with sample 1A leaching up to two times, and sample 4Q up to four times, more than the other samples. Mg, Si, Al and Fe were all at 0.30% or less, with Fe being the least abundant element. The indicators used for assessing the dissolution of undesired species, Si, Al and Fe yield, all show similar patterns with decreased pH. That is, small increase between 0.05 M and 0.11 M, corresponding to pH 3.7-3.3. Significant Si, Al and Fe yield occurred at pH < 3.2 , corresponding to the 0.22 M solution. In the case of ^9Be and ^{10}Be , however, the difference between the elemental yield of 0.05 M and 0.11 M was significant, in some cases with a twofold increase in yield between the two concentrations. Solution pH between 3.2-3.5, corresponding to a concentration of 0.11M for most samples, maximally desorbed ^{10}Be without significantly affecting other grain components. Decreased pH/increased solution concentration had less effect on ^{10}Be yield from samples 1A and 4Q, which had the lowest average ^{10}Be content of all samples.

Figure 10. pH levels of 0.05 M, 0.11 M, 0.22 M and 0.44M acetic acid during the 16-hour experimental leach. A. pH levels of solution blanks through the 16-hour pH observations showing lower pH with increased solution molarity. B. Average sample solution pH for all acid concentrations during the 16-hour acetic acid leach, showing more stability of pH and smaller errors with increased solution molarity. Error bars represent the standard deviation of each solution molarity. C. pH measurements of all sample solutions, showing the trend of decreased pH level with increased acid concentrations and the stabilization of pH after 4 hours of leaching. Sample 1A, a calcite-rich sample, is an outlier which was measured at higher pH than the rest of the samples within each solution concentration. This difference between sample 1A pH and the rest of the samples decreased with increased solution concentration. D. The distribution of pH measurements of all samples between the four different solution concentrations. See Tables A1-A4 in appendix A for data.

Figure 11. Elemental yield as a function of pH at hour 4 of the acetic acid extractions, showing increased yield with decreased pH and the ideal range for maximum desorption of the elements without significantly affecting Si, Al and Fe (shaded areas). The effects of relatively high Ca content on the pH can be seen with samples 1A and 4Q. Ca, Mg, Si, Al and Fe are in percent sample mass, ^9Be is in parts per million of sample mass and ^{10}Be is in 10^7 atoms g^{-1} . Colors indicate solution concentration and shapes indicate the individual samples. ^{10}Be data for 0E & 1B is from the 1st method. See Table A5 in appendix A for data.

4.2.2. High pH Sodium Carbonate Extraction

A high-pH extraction was performed to investigate whether amorphous silica phases, which are insoluble at $\text{pH} < 9$ (Takeno, 2005), host significant amounts of meteoric ^{10}Be in the samples. Solution pH of the 2 M Na_2CO_3 was measured at 11.43, in the range at which amorphous silica is soluble. ICP-OES analysis was challenging due to very low abundances of the elements that were measured. The lower limit of detection (LLD) was calculated for each element based on the regression of the calibration curve. Elemental yield from the extraction was below the LLD for most elements, including Si. Fe was the only element detected, at 0.003% of sample weight, which was at the LLD for Fe in the analysis. Meteoric ^{10}Be from the extraction was only about 2 times as high as the 0.007×10^7 atoms in the solution blank (Table 2).

Table 5. The mass of leached elements and ^{10}Be concentrations in the Na_2CO_3 leachates.

	Mg	Ca	Fe	Al	Si	^9Be	$^{10}\text{Be} (\times 10^7 \text{ atoms g}^{-1})$
0A	< 0.008	< 0.023	0.003	< 0.002	< 0.166	< 0.003	0.015
0B	< 0.008	< 0.023	0.003	< 0.002	< 0.166	< 0.003	0.019
1A	< 0.008	< 0.023	0.003	< 0.002	< 0.166	< 0.003	0.016

4.2.3. Effects of 0.5 M HCl on Clay Minerals

Methods 1 and 2 were designed to study the effect of 0.5 M HCl on clay minerals. Two aliquots of each sample were treated with identical procedures except for the timing of clay separation which was varied to be after the HCl leach (method 1) and before the HCl leach (method 2). More ^{10}Be was measured in the residual fraction (non-clay silicates) in all samples from method 2, making the oxyhydroxide-bound ^{10}Be a smaller fraction relative to method 1 (Fig. 12). XRD patterns of the smectite clays in the samples show the unintended dissolution of smectite by HCl (Fig. 13). Therefore, some clay-bound ^{10}Be released in the HCl extraction artificially over-counted the oxyhydroxide-bound ^{10}Be . The adsorbed ^{10}Be was measured in the same quantities regardless of which method was used and was the second most ^{10}Be -rich fraction in both methods. The clay fraction was larger from method 2 in samples 1B and 0B. Lower total ^{10}Be , as well as lower clay- ^{10}Be , in sample 0E from method 2 might be due to heterogeneity of the sample as it was initially subsampled from the storage bag before analysis.

Figure 12. ^{10}Be yield from samples from methods 1 and 2, showing the effects of unintended dissolution of clays when targeting amorphous fractions if the clay fraction is not separated from the samples before the HCl extraction. Clays were separated after the HCl extraction in method 1 (1) and before the HCl extraction in method 2 (2). See Table A5 in appendix A for data.

Figure 13. Smectite peaks from X-ray diffraction patterns of the clay sized particles in sample 0B show the effects of 0.11 M acetic acid (blue, $2\theta = 6.28$) and 0.5 M HCl (red, $2\theta = 6.3$) on the smectite clays in the sample. Black peak represents untreated sample ($2\theta = 6.1$). All peaks rescaled for peak comparison. All other samples showed similar trends.

4.2.4. Grain Size Dependency of Meteoric ^{10}Be Fractionation

Grain size separations of the samples were performed in method 3 prior to the chemical extractions to investigate the effects of grain size on ^{10}Be fractionation. Prior to separating the samples into medium sand, fine sand, silt, coarse clay, and fine clay, the samples were rigorously mixed to disaggregate fine particles apart from larger particles and from one another. The relative phase fractionation between the samples differed substantially depending on which method the samples were subject to (Table 3, Fig. 14). Far more ^{10}Be was measured in the clay lattice fraction in samples from method 3, 35-60%, than from methods 1 and 2, 5-12%. Oxyhydroxide and residual fractionation was larger in methods 1 and 2 than in method 3. Adsorbed ^{10}Be was relatively comparable between all methods, although a larger variation was found between the samples from method 3.

Figure 14. Relative phase fractionation of total ^{10}Be concentrations of all samples shows a distinct difference between methods 1 and 2, and method 3. Far more ^{10}Be was measured in the clay fraction in the samples that were subject to method 3. Methods are indicated in parentheses next to the sample name. See Tables A6 and A7 in appendix A for data.

Table 8. Summary of the fractionation of ^{10}Be in the samples between the four sediment phases.

	Adsorbed	Oxyhydroxides	Residual	Clay lattices	Total ^{10}Be ($\times 10^7$ atoms g^{-1})
1B (1)	0.56	0.96	0.28	0.14	1.94
1B (2)	0.50	0.75	0.33	0.22	1.79
0E (1)	2.70	7.10	1.04	1.09	11.93
0E (2)	2.69	4.63	1.55	0.52	9.39
0B (1)	6.38	13.42	1.58	1.00	22.39
0B (2)	6.46	10.85	2.53	2.32	22.17
0B (3)	10.40	7.06	0.63	14.53	32.61
1A (3)	2.60	3.33	0.46	3.42	9.82
0A (3)	4.91	0.53	0.22	8.47	14.13
4Q (3)	22.75	47.20	4.75	58.51	133.22

Parentheses indicate which method the sample was treated with 1) Method 1, (2) Method 2, and (3) Method 3.

Sample 0B, the control sample, which was subject to all three methods, shows the effects that the different methods have on the ultimate ^{10}Be yield and the relative fractionation within the sample. The main difference was manifested as a 50% increase in total ^{10}Be yield from method 3 relative to methods 1 and 2. Compared to the average of ^{10}Be concentrations in each fraction of methods 1 and 2, 60% more ^{10}Be was in the adsorbed fraction in method 3. Oxyhydroxide-bound (HCl-extracted) and residual ^{10}Be was 42% and 70% less in method 3, respectively, than in methods 1 and 2. Clay-bound ^{10}Be increased by 777% in method 3 from methods 1 and 2. Such difference in total beryllium concentrations from the same sample after similar extraction and digestion methods, suggests that the initial sample preparation played a significant role in the process, most notably with the increased inclusion of clay-sized particles (Fig. 15).

Two samples, 0E and 1B, were only subject to methods 1 and 2. The ^{10}Be fractionation of those samples was normalized to method 3 to allow for a more accurate analysis of the subglacial chemical processes at the study site, since method 3 appeared to better reflect the ^{10}Be fractionation in the samples (Fig. 15).

Figure 15. ¹⁰Be yield from the three different methods (indicated in parentheses) varied significantly, with similar total ¹⁰Be from methods 1 and 2 but different relative proportions of the adsorbed, oxyhydroxides, residual and clay lattices fractions. Method 3 yielded 46% more total ¹⁰Be, with the proportions of adsorbed, oxyhydroxides and residual and clay lattice fractionation significantly different from methods 1 and 2. Results from method 3 are only displayed for the < 63 μm size fractions. See Table A6 in appendix A for data.

Fractionation of meteoric ¹⁰Be in all samples shows that weathering products are the primary hosts of ¹⁰Be in the samples (Table 4, Fig. 16). Meteoric ¹⁰Be concentrations of 2 - 22 × 10⁷ atoms g⁻¹ in the oxyhydroxide and clay fractions of the freshly emerging, subglacially-derived sediments are strong evidence for subglacial chemical weathering in the catchment of Law Glacier. That is 53-84% of the total ¹⁰Be are fractionated in weathering products (Fig. 17). Fractionation of ¹⁰Be between adsorbed, oxyhydroxide, residual and clay lattice sediment fractions shows that clays were the primary host of ¹⁰Be in the sediments. Clay-bound ¹⁰Be was highest in sample 0A at 60% of the total ¹⁰Be, while it was lowest in sample 1A at 35% of the total sample ¹⁰Be. The second ¹⁰Be-rich fraction in the freshly emerging samples is the adsorbed, at 27-35% of the total sample ¹⁰Be. In the long-exposed sample, oxyhydroxide concentrations were the second most ¹⁰Be-rich fraction, accounting for 35% of the total. The oxyhydroxide-bound ¹⁰Be content varied between 4% and 34% in the freshly emerging samples, lowest in sample 1A. Residual ¹⁰Be was consistently the smallest fraction in all samples (Table 4, Fig. 16).

Table 4. Meteoric ^{10}Be concentrations measured in all sediment phases.

	Adsorbed	Oxyhydroxides	Residual	Clay lattices	Total ^{10}Be ($\times 10^7$ atoms g^{-1})
0A	4.91	0.53	0.22	8.47	14.13
0B	10.40	7.06	0.63	14.53	32.61
0E	4.36	3.41	0.39	7.05	15.21
1A	2.60	3.33	0.46	3.42	9.82
1B	0.86	0.50	0.09	1.55	3.00
4Q	22.75	47.20	4.75	58.51	133.22

Figure 16. Clays are the primary ^{10}Be hosts in the freshly emerging sediments at Mt. Achnar Moraine. Top insert includes sample 4Q. All samples normalized for the difference between the average of methods 1 and 2, and their difference from method 3. See Table A6 and A7 in appendix A for data.

Figure 17. Meteoric ^{10}Be concentrations in freshly emerging subglacial sediments (0A, 0B and 1A) are grain size dependent, i.e. more ^{10}Be accumulates in smaller grain sizes than in larger grain sizes (larger, orange pie charts). Phase fractionation of ^{10}Be (smaller pie charts) in the residual grain sizes (medium sand, fine sand, and silt) varies, with adsorbed species being the dominant ^{10}Be phase in samples 0A and 0B, but oxyhydroxides more dominant in the calcite-rich sample 1A and the sample with a 5×10^5 year exposure history, 4Q. In the clay-sized fraction, clay lattices are the dominant ^{10}Be hosts over adsorbed species in all cases. ^{10}Be was only measured in the medium sand fraction in sample 0B. See Tables A10 and A11 in appendix A for data.

Meteoric ^{10}Be concentrations in the freshly emerging sediments at the Mt. Achnar Moraine were dependent on grain size, i.e., the smaller the grains, the higher the ^{10}Be concentrations. Clay and silt-sized particles contained 89-95% of the total ^{10}Be in the samples. Clays were the most ^{10}Be -rich size fraction in all cases, at 53-84%. The relative size between coarse clay (settled in 15 hours) and fine clay (still in suspension after 15 hours) did not appear to matter, since the relative fractionation of ^{10}Be between the two differs between samples. Of the clay-sized particles, clay lattices were the primary host of ^{10}Be over the adsorbed fraction. Medium sand was the sparsest ^{10}Be fraction in the freshly emerging samples, while in sample 4Q, the fine clay fraction contained the least ^{10}Be . The adsorbed fraction is the dominant ^{10}Be fraction of the non-clay sized particles in samples 0A and 0B. In samples 1B and 4Q, the fractionation of ^{10}Be in the non-clay sized particles was dominated by HCl-extractable ^{10}Be (Fig. 17).

A strong correlation ($R = 0.96$, $P = 0.004$) between smectite and ^{10}Be concentrations in the samples suggests that smectite is the primary clay-host of ^{10}Be in the samples and is therefore strong evidence for authigenic clay formation in the subglacial environment of Law Glacier (Fig. 18). Kaolinite, the other abundant weathering product at the moraine (Graly et al., 2020), showed no significant correlation with ^{10}Be concentrations. Chlorite and illite likewise are not correlated with ^{10}Be in the samples.

Figure 18. - The most abundant weathering products at the Mt. Achnar Moraine, smectite clays, have a strong correlation with meteoric ^{10}Be concentrations in the samples. R and P values of all the clay species included in the legend. See Table A12 in appendix A for data.

4.2.5. Chemical Controls on ^{10}Be Fractionation

To determine whether the chemical composition of the samples exerted significant controls on ^{10}Be accumulation, each leachate from all extractions was analyzed with ICP-OES. The biggest difference in elemental yield between the three methods was in the acetic acid leach, where carbonates and adsorbed species were the targeted phases. ^9Be yield from methods 1 and 2 was 1-2 orders of magnitude higher in every extraction than in method 3.

In the acetic acid leach, Ca was the most abundant element, suggesting that carbonates were being dissolved preferentially over other minerals. That's likely because this is a weak acid and carbonates are the most likely to dissolve. Samples from method 3 yielded more of every element, while yield from methods 1 and 2 were almost identical for each element. No Fe was detected in the acetic acid leachates from methods 1 and 2. Yields in all other extractions were similar between the methods, except for ^9Be , and Al in the totally digested fractions. Sample 4Q leached Mg, Ca, Na, K, Fe and Al in higher quantities than all other samples (Fig. 19).

Oxyhydroxides were targeted with a 0.5 M HCl extraction, which yielded similar concentrations of all elements regardless of which method was used, with method 3 just slightly higher on average. Between methods 1 and 2, yields were slightly higher for all elements from method 2, where the clays were separated before the HCl extraction. Fe, Al and Si were the most abundant elements at 0.4-1% sample mass. Ca was relatively abundant as well, ranging from 0.3 to 0.8% sample mass, highest in the most calcite-rich samples (1A, 1B and 4Q). This suggests that some of the carbonates withstood the acetic acid extraction and were further dissolved in the HCl extraction. Sample 4Q yielded more Mg, Na and Fe than all other samples (Fig. 19).

Total dissolution of the samples was not successful in any case except for the fine clay fraction of sample 4Q. Some residue in all other samples in all fractions withstood the total digestion. Totally digested non-clay particles make up the residual, where Fe and Al were the most abundant elements at 1.3-2.4% and 2.0-4.8% respectively. Al yield was higher from method 3 than methods 1 and 2. Similar elemental yield patterns can be seen for the totally digested clay lattice fraction of the samples, in slightly higher quantities. Fe and Al yields were at 7.4-16.1% and 3.0-20.3% respectively, with more Al from method

3 than methods 1 and 2. Methods 1 and 2 yielded less Al in the residual and clay lattice fractions than method 3. More Mg, Ca and Na was leached from 4Q in the total digestion of clay minerals than from all other samples (Fig. 19).

Elements measured in the chemical analyses were examined for statistical correlation with total ^{10}Be concentrations in the samples. Significant statistical correlations do exist but seem to be driven solely by the sample with exposure history at the moraine's surface. When all samples are included in the analysis, 21 instances of significant statistical correlation were found, distributed between all elements except for Fe and Si. Mg demonstrates the strongest statistical correlation with ^{10}Be with a P-value of 0.004, $R = 0.948$ and R^2 of 0.899 in the acetic acid leachates, and $P = 0.002$, $R = 0.962$ and $R^2 = 0.925$ in the HCl leachates. Na had the second strongest correlation. Acetic acid leachates had the strongest correlation with ^{10}Be content out of all leachates, followed by HCl leachates (Table 5). This analysis was also done on the freshly emerging samples only, excluding sample 4Q. The number of significant statistical correlation between individual elements and ^{10}Be dropped to only one instance: $R = 0.832$ in the case of K. Correlations of ^{10}Be concentrations with individual elements therefore do not seem to apply within the freshly emerging subglacial sediments (Table A13, appendix A). Sample 4Q leached more Mg, Ca, Na, K, Fe and Al than all other samples.

Figure 19. Elemental yield of all extractions from methods 1 (purple), 2 (blue) and 3 (green) shows a distinct difference between methods 1 and 2, and method 3 in the acetic acid-extractable species and in Al yield in both totally digested fractions. Elemental yield in the HCl-extractions was similar between all methods. Sample 4Q most frequently had the highest elemental yield out of all the samples. Acetic acid-extractable species are interpreted as carbonates (Cb) and adsorbed species (top left), HCl-extracted species are interpreted as oxyhydroxide-bound (top right), totally digested non-clay silicates are interpreted as residual (bottom left) and totally dissolved clay-sized particles are clay minerals (bottom right). Na was not detected in totally digested fractions in methods 1 and 2. See Table A14 in appendix A for data.

Table 5. Summary of correlation coefficients and significance of correlation (P, R and R² values) for total ¹⁰Be concentrations and elemental extracts (in % sample mass) from the < 63 µm fraction of the samples.

	Mg	Ca	Na	K	Fe	Al	Si	⁹ Be (ppm)	¹⁰ Be (×10 ⁷ atoms g ⁻¹)
<i>Acetic Acid Extracts</i>									
0A	0.08	1.68	0.02	0.04	0.01	0.03	0.50	0.57	3.22
0B	0.10	1.77	0.03	0.04	0.01	0.02	0.54	0.64	5.33
0E	0.03	0.42	0.01	0.01	0.00	0.00	0.12	-	4.36
1A	0.10	2.76	0.02	0.07	0.01	0.03	0.59	0.90	1.70
1B	0.01	0.45	0.01	0.01	0.00	0.01	0.10	-	0.86
4Q	1.29	8.00	10.09	0.32	0.03	0.14	0.18	0.17	14.53
P-value	0.004	0.016	0.005	0.008	0.018	0.009	0.628	0.653	
R	0.948	0.893	0.943	0.925	0.887	0.920	0.253	0.235	
R ²	0.899	0.798	0.890	0.856	0.786	0.847	0.064	0.055	
<i>HCl Extracts</i>									
0A	0.08	0.25	0.01	0.03	0.58	0.52	0.54	-	0.53
0B	0.11	0.42	0.01	0.04	0.42	0.64	0.65	0.32	1.35
0E	0.14	0.50	0.01	0.05	0.42	0.69	0.60	-	3.41
1A	0.12	0.77	-	0.05	0.69	0.77	0.94	0.45	1.22
1B	0.12	0.42	0.01	0.04	0.39	0.56	0.57	-	0.50
4Q	0.24	0.50	0.03	0.05	0.93	0.69	0.84	0.41	12.25
P-value	0.002	0.796	0.011	0.244	0.070	0.499	0.381	0.376	
R	0.962	0.137	0.913	0.564	0.776	0.348	0.441	0.446	
R ²	0.925	0.019	0.834	0.318	0.602	0.121	0.195	0.199	
<i>Totally Digested Residual Particles</i>									
0A	0.01	0.03	-	2.37	2.87	4.72	-	-	0.14
0B	0.91	0.27	0.99	2.36	3.04	7.04	-	-	0.32
0E	0.04	0.25	-	1.70	2.08	2.72	-	-	0.39
1A	-	0.02	0.68	1.68	2.56	4.11	-	0.23	0.30
1B	-	0.42	-	1.69	3.08	2.54	-	-	0.09
4Q	0.04	0.04	0.56	1.96	3.13	4.78	-	0.66	3.04
P-value	0.796	0.431	0.957	0.983	0.510	0.767		0.005	
R	0.137	0.401	0.029	0.012	0.340	0.157		0.942	
R ²	0.019	0.160	0.001	0.000	0.116	0.025		0.887	
<i>Totally Digested Clay Particles</i>									
0A	0.57	0.36	0.53	2.44	8.15	13.82	-	3.07	8.47
0B	0.13	0.11	0.34	1.75	8.26	9.89	-	2.60	7.44
0E	0.43	0.68	-	3.21	11.79	6.52	-	-	7.05
1A	2.25	0.60	0.58	4.89	9.32	20.29	-	4.10	2.23
1B	0.29	0.79	-	2.87	9.76	5.10	-	-	1.55
4Q	3.06	2.29	1.27	5.27	12.51	17.38	-	-	37.37
P-value	0.127	0.021	0.034	0.268	0.168	0.440		0.428	
R	0.692	0.881	0.846	0.541	0.643	0.393		0.403	
R ²	0.479	0.775	0.715	0.292	0.414	0.155		0.163	

Statistically significant values are shown in bold.

Values for leached elements from samples 0E and 1B are the averages of the elemental yield from methods 1 and 2. ¹⁰Be values of those samples have also been normalized to method 3 (see section 4.2.4.).

Dashes (-) represent instances where data were not usable due to calibration issues.

5. Discussion

5.1. Sample Mineralogy

Beryllium is known to adsorb to the surfaces of fine-grained sediment particles in aqueous systems, precipitate with amorphous material, in particular HCl-extractable grain coatings, and be incorporated into clay minerals during chemical weathering (Barg et al., 1997; McHargue & Damon, 1991; Singleton et al., 2016). Mineralogical data was investigated in order to assess potential hosts for meteoric ^{10}Be in the $< 63\ \mu\text{m}$ fraction of the samples. Clays and amorphous, non-crystalline materials made up about half of the samples in weight, suggesting an array of potential ^{10}Be hosts in the samples (Table 1). Knowing the mineralogical properties of the samples can help with understanding the effects of the chemical procedures which the samples are subject to, and to determine if there are certain mineralogical characteristics which control ^{10}Be retention in the sediments.

Illite, chlorite, kaolinite and smectite were the four main clay minerals detected in the samples. A mineralogical mass balance study, where the sediments at the moraine were compared to the source rock, found a 15-fold increase in smectite and 7-fold increase in kaolinite in the sediments relative to the source rock (Graly et al., 2020). This strongly suggests authigenic kaolinite and smectite clay formation in the subglacial environment, making kaolinite and smectite potential hosts of meteoric ^{10}Be . Feldspars and pyroxenes are the main sources of cations on the Mt. Achnar subglacial system (Graly et al., 2020). Feldspars were far more abundant, suggesting that Al-bearing smectites, e.g. montmorillonite, might be more abundant than Fe and Mg-bearing smectites like saponite. Saponites are trioctahedral, bearing divalent cations in their octahedral sites (e.g. Mg^{2+} , Fe^{2+}) (Velde, 2013). Montmorillonite and kaolinite clays are dioctahedral, meaning that their octahedral sheets bear trivalent cations (e.g. Al^{3+}) in their octahedral sites (Velde, 2013). However, beryllium exists exclusively in trivalent position in silicate minerals and is known to cause structural collapse if substituted into octahedral sites (Hawthorne & Huminicki, 2002; Wang et al., 2017). It is therefore expected that the meteoric ^{10}Be occupies tetrahedral positions within the clay lattices in the samples. The difference between smectites and kaolinite is that smectites are 2:1 clay minerals, with 2

tetrahedral sheets for each octahedral sheet in their structure, while kaolinite is a 1:1 clay (Velde, 2013). Having twice the amount of potential tetrahedral sites for ^{10}Be to occupy, smectite clays have, in theory, the capacity to carry larger quantities of the nuclide. The strong correlation between ^{10}Be and smectite abundance in the sample, and the lack of correlation with all other clay minerals, strongly suggests that authigenic smectites are effective scavengers of meteoric ^{10}Be .

5.2. Experimental Development

5.2.1. pH Dependency of Element Adsorption/Desorption

The aim of the analytical techniques described here is to evaluate the amount of ^{10}Be adsorbed on the surface of mineral grains. ^{10}Be exists adsorbed to fine-grained particles in solution at $\text{pH} < 6$ (You et al., 1989). The pH experiment was designed to assess the conditions under which the yield of adsorbed elements was maximized, especially ^{10}Be , while affecting other grain components minimally. The acetic acid extractions were tested to ensure that other components such as oxides and oxyhydroxides, and clay minerals were not extracted. Acetic acid extractions were used to both dissolve carbonates and desorb adsorbed species. Therefore, Ca and Mg released into solution could not be used as indicators for the dissolution of undesired phases due to their expected dissolution from carbonates. Si, Al and Fe were used as indicators for the undesired dissolution of grain coatings and clays.

Stabilization of sample solution pH after 4 hours of leaching suggests that cation exchange between the acid and the samples was exhausted in approximately that time (Fig. 10). All sample solution pH measurements were in the range where beryllium exists in solution and increasingly, exponentially, adsorbs to fine particles with increased pH (Fig. 1) (You et al., 1989). ^{10}Be data from three samples subjected to the pH experiments are in agreement with previous studies, showing increased yield/desorption of ^{10}Be with decreased pH.

For the intended purposes of this study, a concentration of 0.11 M acetic acid is ideal to achieve maximal desorption of ^{10}Be and other adsorbed species while minimally affecting amorphous grain components and aluminosilicates. The 0.11 M sample solutions remained at a pH that yielded significantly more ^{10}Be than the 0.05 M solution

and did not reach the pH 3.2 threshold at which Si, Al and Fe yield significantly increases. Silica desorption did not occur at the pH levels of this experiment since amorphous silica phases are insoluble at $\text{pH} < 9$ (Takeno, 2005). Aluminum exists exclusively as the Al^{3+} ion at $\text{pH} < 4$, but its abundance as a free ion, available for adsorption in natural systems is vanishingly small due to its high charge-to-radius ratio (Harris et al., 1996; Rumble, 2020). Therefore, Si and Al detected in the leachates indicate the undesired dissolution of more immobile phases, e.g. amorphous oxides or aluminosilicates, at $\text{pH} < 3.2$.

Ca content in the samples is a potential contributing factor to adsorbed ^{10}Be yield, preventing the desorption of ^{10}Be (and other adsorbed species) by consuming the majority of the H^+ ions available for exchange from the acid. Acetic acid primarily targets CaCO_3 , which Be is unable to complex with (Southon et al., 1987). In that case, Ca-rich samples might need higher concentrations of acetic acid to ensure that the pH stays within the 3.5-3.2 window in order to desorb all the available adsorbed ^{10}Be . This effect can be seen on the calcite-rich samples, 1A and 4Q. All samples were measured at a small range of pH throughout the experiment, except 1A, which was always at significantly higher pH (Fig. 10C). The significance of this effect decreases with increased solution concentration (Fig. 10D).

5.2.2. High pH Extraction Using Sodium Carbonate (Na_2CO_3)

A high (> 10) pH extraction was performed to examine whether the excess of weathered silica in the weathering products at the moraine formed amorphous silica phases which ^{10}Be complexes with. ICP-OES analysis was challenging due to the low levels of elements in the sample leachates. Fe was the only element detected in all samples, at 0.003% of the sample mass, while all other elements were below the lower limit of detection (Table 2). The extraction was designed to specifically target Si-complexes, therefore it is curious that silica was not detected in any of the samples. This suggests that the silica is complexed in phases that are insoluble at pH 10.4. ^{10}Be yield from the extractions was insignificant relative to all other extractions in this study, at levels only twice the concentration of the solution blank. While it is possible that the extraction did not dissolve any sediment phases, which would explain the low elemental

abundances in the leachates, it is also possible that an instrument with a higher level of sensitivity is required (i.e. ICP-MS) to produce more meaningful results.

5.2.3. Effects of 0.5 M HCl on Clay Minerals

The goal of testing the effects of the 0.5 M HCl extraction on the clays was to assess the importance of clay separations when targeting the amorphous oxyhydroxide fraction of the sediment grains. If clay minerals were dissolved in the HCl leach, that would cause a mis-estimation of ^{10}Be released from clays as ^{10}Be released from oxyhydroxides. Clay-bound ^{10}Be was measured in larger quantities in samples subject to method 2, where the clays were separated from the samples before the HCl extraction and were therefore not included in the HCl extraction. In method 1, the clays were separated after the HCl leach and were unintentionally dissolved. Evidence of this can be seen in the XRD patterns of the clays before and after the acetic acid and HCl extractions. A shift in the 2θ position of the smectite peaks, from 6.1° 2θ of the untreated sample to 6.6° 2θ after the HCl leach indicates the partial dissolution of pure smectite (Fig. 13). This would explain the larger quantities of ^{10}Be in the oxyhydroxide phase in method 1, i.e. ^{10}Be leached from dissolved clays in the HCl leach were incorrectly interpreted as oxyhydroxide-bound ^{10}Be (Fig. 12). This effect underlines the significance of clays as ^{10}Be hosts.

XRD patterns of the smectite clays before and after the acetic acid and HCl extractions show that both extractions affect the structure of the clay to varying degrees. A shift of 0.2° 2θ from the untreated to the acetic acid treated sample might indicate that a cation exchange took place during the acetic acid extraction. The extraction targeted and released Ca into solution, which potentially exchanged for K in the clay structure, causing a shift in the peak position (Chipera et al., 1995). pH levels were not measured in this particular acetic acid leach of the clays before XRD analysis. Therefore, unintended dissolution of some clay minerals cannot be ruled out. HCl had a greater effect on the smectites, shifting the peak by 0.6° 2θ from the untreated sample. Smectite is commonly interlayered with illite, in which case the XRD peak becomes relatively broadened to the right (higher 2θ values) (Drits et al., 1998). The pattern for the HCl-treated smectites shows such broadening, indicating dissolution of pure smectite, making the interstratified illite-smectite signal more prominent (Fig. 13).

5.2.4. Grain Size Dependency of ^{10}Be Fractionation

The study of the effects of grain size on ^{10}Be retention in the samples revealed that ^{10}Be concentrations in the samples were highly dependent on grain size, consistent with previous studies (Boschi & Willenbring, 2021; Singleton et al., 2016; You et al., 1989) and further underscoring the importance of clays on ^{10}Be retention. The most significant difference between the methods was the initial preparation of the samples in method 3 before grain size separations and extractions. Before the samples were separated into the five different grain size fractions, the samples were subject to vigorous disaggregation by mixing in a blender and sonication. This was done in order to make sure that clay-sized particles encrusted onto larger silt and sand grains, were not left out of the analysis when the larger grains were sieved out prior to analysis. In the case of method 3, 50% more ^{10}Be was measured in sample 0B compared to when it was subject to methods 1 and 2 (Fig. 15). This also affected the relative phase fractionation of ^{10}Be in the samples greatly, making clays the dominant ^{10}Be host in method 3 as opposed to oxyhydroxides (HCl-extracted species) in methods 1 and 2. Method 3 is most likely to accurately represent the fractionation of ^{10}Be between the sample phases.

Higher Al yields in the clay dissolution from method 3 suggests that clays were included in higher quantities in the extractions than in methods 1 and 2. If that is the case, then it's possible that more montmorillonite (Al-smectite) and/or illite was present in the clay fraction in method 3 than in methods 1 and 2, which might have unintentionally dissolved a portion of those clay minerals in the HCl leach (Fig. 19).

In the samples included in method 3, clay-sized particles were the dominant hosts of ^{10}Be . The relative size of the clay particles, that is the proportion of fine and coarse clay fractions, does not seem to be a major control on ^{10}Be retention. The majority of the ^{10}Be in the clay-sized fractions comes from the total dissolution of the clay minerals, i.e. from within the clay lattices, as opposed to being adsorbed to the clay surfaces. This holds true for the freshly emerging sediments as well as sample 4Q. In the larger grain sizes, silt, fine sand and medium sand, the phase fractionation differs between the freshly emerging sediments and the calcite-rich sample, 1A, and sample 4Q (Fig. 17). Adsorbed ^{10}Be was most abundant, followed by HCl-extracted ^{10}Be (oxyhydroxides), in samples 0A and 0B, while the order was reversed in 1A and 4Q (Fig. 17). The potential cause for

this effect may be the buffering of the acetic acid extraction by Ca (Fig. 10C). In the case of the carbonate-rich samples, in which the pH level was a little higher, some adsorbed species seemed to withstand the acetic acid extraction and be leached into solution in the HCl extraction instead. Therefore, some of the ^{10}Be in the oxyhydroxide (HCl-extracted) and clay fractions in samples 1A and 4Q might be adsorbed ^{10}Be .

Meteoric ^{10}Be is unlikely to have been inherited from the source rock due to the > 30 Ma ice cover at the site and the 1.39×10^6 years half-life of the nuclide (DeConto & Pollard, 2003; Korschinek et al., 2010). Atmospherically deposited meteoric ^{10}Be is unlikely to be a significant factor due to the relatively short surface exposure of the freshly emerging sediments ($< 10 \times 10^3$ years). In-situ ^{10}Be production rates are on the order of $3.60\text{--}3.82$ atoms $\text{g}^{-1}_{\text{quartz}} \text{yr}^{-1}$ (Kaplan et al., 2011), and would only be measured in totally digested quartz minerals. In-situ ^{10}Be is therefore unlikely to contribute significantly to the concentrations measured in the samples. Residual ^{10}Be in the samples can therefore mean two things. First, the substitution of Al and/or Si by ^{10}Be into tetrahedral sites in primary minerals. These silicates would then have withstood further disintegration during entrainment and transport to the surface, and the acetic acid and HCl extractions. No mention of this could be found in the literature. Second, the ^{10}Be measured in the residual fraction was from resistant oxides and/or clay particles that were not successfully separated from the residual particles and withstood the acetic acid and HCl extractions. The latter is more likely, since the incorporation of ^{10}Be into authigenic secondary mineral phases formed during weathering is known to occur (Barg et al., 1997; You et al., 1989).

5.2.5. Chemical Controls on ^{10}Be Fractionation

All leachates from the chemical extractions of the samples were chemically analyzed with ICP-OES to study the effects of the extractions and whether particular elements were correlated with ^{10}Be in the samples (Fig. 19). Acetic acid extractions were used to dissolve carbonates and adsorbed species in the samples due to beryllium's adsorption behavior in aqueous solution. Mg and Ca are species associated with carbonates, which in the case of these samples would be calcite and dolomite (Table 1). Although some Mg and Ca might have been adsorbed to grain surfaces. Higher yields of all elements from the samples by method 3 is most likely due to the disaggregation of the

sample. It is likely that clay-sized particles in methods 1 and 2 were unintentionally left out of the extractions due to them adhering to the surfaces of larger grains which were sieved out before the chemical extractions. The mixing and sonicating prevented major aggregation and the undesired exclusion of clay-sized particles from the leach due to the removal of larger grains, to which they adhered, in the initial sample preparation. Clay-sized particles play a major role in sediment adsorption due to their large surface areas and surface charge, which attract ions from solution (Velde, 2013). Samples in method 3 included more clay-sized particles which could explain the higher yields of desorbed species. Aggregation of clay-sized particles could also prevent desorption of some adsorbed elements, as the acid would only affect the outer-surface of the aggregates, leaving adsorbed species within aggregates unaffected.

The base cations, Ca^{2+} , Mg^{2+} , Na^+ and K^+ were expected to be the main species desorbing in the acetic acid leach (Rayment & Higginson, 1992). It is therefore curious that Fe, Al and Si were present in the leachate. Al^{3+} can exist as a mobile cation in acidic (pH 3-4) environments, but, like Fe, is more commonly bound in oxides or oxyhydroxides (Drever, 1997; Stumm et al., 1996). Graly et al. (2020) reported a decline in magnetic susceptibility in the moraine sediments relative to the source rock, indicating oxidizing conditions in the Mt. Achnar subglacial system. Soluble Fe^{2+} is therefore unlikely to be abundant in the system. The presence of Fe, Al and Si might therefore indicate the unintended dissolution of amorphous phases or clay particles in small quantities in the weak acid, especially in clay-sized fractions from method 3.

^{10}Be is unable to fractionate with carbonates (Southon et al., 1987) and therefore no ^{10}Be is expected to be associated with the dissolution of those species. However, the presence of carbonates seems to have buffered the pH of the acetic acid, making it less effective in desorbing adsorbed species in calcite-rich samples (Fig. 11). That might explain the presence of Mg and Ca in the HCl extraction, as carbonates that withstood the acetic acid extraction, e.g., dolomite. Other possible Mg and Ca sources are zeolites and phosphates, whose abundances were below XRD detection, but were reported in the clay mounts in Graly et al. (2020).

Oxides and oxyhydroxides were the targeted phases in the HCl extraction, although Si yield suggests dissolution of other phases. XRD patterns show the dissolution

of aluminosilicates in methods 1 and 2, but the clay fractions were excluded from the HCl extraction in method 3 (Fig. 13). Therefore, similar Si yields between all methods was surprising. Amorphous silica dissolution is unlikely due to the insolubility of amorphous Si compounds at $\text{pH} < 9$. The only soluble Si phase at $\text{pH} < 7$ is silicic acid ($\text{Si}(\text{OH})_4$) (Queneau & Berthold, 1986). As was mentioned in the discussion about ^{10}Be in the residual grains, it is possible that some clay particles were not successfully separated from surfaces of residual grains in method 3. Those clays would therefore have been subject to the HCl extraction along with the residual grains and dissolved, resulting in the release of Si into the leachate. The allophane and phosphate content reported in Graly et al. (2020) are potential sources of Si and Al.

The ratio of meteoric ^{10}Be and ^9Be could be used to infer the ratio of detrital vs. authigenic clays in the samples. ^9Be , the stable isotope of beryllium, is of detrital origin, i.e. inherited from the source rock. Inherited clays, e.g., illite, would therefore have a higher $^9\text{Be}/^{10}\text{Be}$ ratio than authigenic clays like smectite, which are more likely to contain meteoric ^{10}Be due to incorporation during neoformation. However, higher quantities ^9Be from methods 1 and 2 in all extractions is unlikely to reflect differences between residual and authigenic clay contents of the samples. Although calibration for the element was always successful, concentrations detected during ICP-OES analysis were only slightly above blank levels, and instrument drift had a significant impact on sample measurements. Therefore, the ^9Be concentrations can vary greatly between analyses without significant differences in sample concentrations and can therefore not be used to infer $^9\text{Be}/^{10}\text{Be}$ ratios.

Total dissolution of residual and clay particles was not successful in any case except for the fine clay fraction of sample 4Q. Therefore, chemical data from ICP-OES analysis of the totally digested fractions does not represent the total chemical composition of the residual and clay particles. Quartz was the most abundant silicate mineral in the samples (Table 1), which could have made up the majority, if not all, of the undissolved residual fraction. Regardless of what minerals withstood the total digestion, meteoric ^{10}Be was detected in the leachates, and therefore the phases with which the ^{10}Be fractionated should be reflected in the leachates. Feldspars and pyroxenes are the main sources of cations in the system at Mt. Achnar moraine (Graly et al., 2020). Based on

the XRD analyses, the dissolved minerals can be inferred from the major cations in the leachates. Mg and Ca in the residual fraction are likely to be sourced from clinopyroxenes (pigeonite and augite), the Na from plagioclase, and K from orthoclase.

Higher Al yields from method 3 in the clay lattice fraction could be attributable in part to the unintended dissolution of clays in the HCl extraction in methods 1 and 2. There are four main clay species in the samples: illite, smectite, kaolinite and chlorite. All exist in the source rock, but the sediments at the moraine contain 15 and 7 times more smectite and kaolinite, respectively, than the source rock (Graly et al., 2020), strongly indicating the two as authigenic weathering products resulting from subglacial chemical processes and candidates for the incorporation of ^{10}Be . The strong correlation between smectite and ^{10}Be ($R = 0.96$, $P = 0.004$) and the weak correlation with kaolinite ($R = 0.28$, $P = 0.363$) shows that smectite is the primary clay-host of meteoric ^{10}Be in the samples.

Sample 4Q reveals what happens to the samples with exposure at the surface of the moraine, as it has been at the moraine surface for 5×10^5 years. A provenance study of the moraine found that the sediments from which sample 4Q was collected had the same source rocks as the freshly emerging samples (Bader et al., 2017). Therefore, a similar mineralogical composition would be expected in terms of the Beacon Supergroup/Ferrar Dolerite source rock ratio. The most notable differences between sample 4Q and the freshly emerging sediments are higher total ^{10}Be concentrations (from surface exposure) (Table 4, Fig. 16), higher quantities of carbonates as suggested by detectable levels of calcite and dolomite (Table 1) and relatively high Mg and Ca content (Table 5), and higher degree of ^{10}Be fractionation in HCl-extractable (oxyhydroxide) phases (Fig. 17). Additionally, zeolites might be present in the sample as shown by higher Al, K, Na and Ca yield in the clay fraction than in the other samples (Fig. 19)(Velde, 2013).

Rough estimation of the expected inventory of sample 4Q after 5×10^5 years at the moraine surface were made using the equation

$$N = q(1 - e^{-\lambda t})/\lambda \quad (1)$$

based on a meteoric ^{10}Be deposition rate (q) of $6.0 \times 10^4 \text{ atoms cm}^{-2} \text{ year}^{-1}$ (Heikkilä, 2007) and the disintegration constant of meteoric ^{10}Be , $5.0 \times 10^{-7} \text{ year}^{-1}$ (Korschinek et

al., 2010). According to these estimations, the calculated sample inventory is $\sim 2.65 \times 10^{10}$ atoms cm^{-2} . Assuming a surface till density of 1.8 g cm^{-3} (Graly et al., 2018a), that's 1.47×10^{10} atoms g^{-1} , an order of magnitude more than the 133.22×10^9 atoms g^{-1} measured in the sample. In-situ ^{10}Be might be a contributing factor in the residual fraction (totally digested quartz minerals), although not significant enough to account for the magnitude of difference. In-situ concentrations measured in other samples with a $\sim 3 \times 10^5$ year exposure on the moraine were on the order of 10^6 atoms $\text{g}^{-1}_{\text{quartz}}$ (Kaplan et al., 2017). Measured concentrations in the residual fraction of sample 4Q were 4.12×10^7 atoms g^{-1} (Table A10), or one order of magnitude higher than should be expected for in-situ ^{10}Be in the fraction. Leaching of ^{10}Be to depth in the Mt. Achernar moraine could potentially cause the maximum concentration where sample 4Q was collected to be higher than what we measured. ^{10}Be is known to leach to depth in soils and the maximum concentrations of soil profiles are often not found at the surface, but rather at depth within the soil profile (Graly et al., 2010). When this is the case, the difference between surface concentrations and maximum concentrations found at depth can be on the order of one magnitude (Graly et al., 2010). This occurs in soil profiles with clay-rich B-horizons where illuviation of ^{10}Be -bearing phases takes place (Graly et al., 2010). Examples of ^{10}Be leaching to depth have been found in soil profiles at Roberts Massif and Thanksgiving Valley in Antarctica, where the maximum ^{10}Be concentrations were at a depth of 5 cm (Diaz et al., 2020). Although the maximum concentrations at 5 cm depth were within the same order of magnitude. The till thickness where sample 4Q was collected is >1 m (Bader et al., 2017), therefore leaching is possible. Percolation of acidic solution through, and weathering activity within, the soil profile are key factors in the leaching of ^{10}Be to depth (Barg et al., 1997). Mineralogical characteristics at depth where sample 4Q was collected are not known and the local climate is extremely dry, making it hard to determine the potential processes of ^{10}Be integration into the profile (Diaz et al., 2020).

Despite the inability of ^{10}Be to fractionate with carbonates, and the lower quantities of smectite in sample 4Q, relative fractionation of ^{10}Be is similar in 4Q as in the freshly emerging sediments (Fig. 14). This indicates similar ^{10}Be behavior, i.e.

fractionation and retention within the samples, in the subglacial environment and at the moraine surface.

Significant statistical correlation between individual elements and ^{10}Be concentrations in the leachates exists in the samples but is entirely driven by sample 4Q, which is the sample richest in ^{10}Be . Most of the correlations are in within the acetic acid and HCl extracts. The correlations are therefore driven by sample 4Q only when the particular elements are leached in the highest quantities in that sample, which also has the highest abundances of ^{10}Be . Lack of statistical correlation within the freshly emerging sediments between ^{10}Be and the elements leached suggests that the chemical composition does not have a strong control over ^{10}Be fractionation (Table A12, Appendix A). The only statistically significant correlation with ^{10}Be concentrations in the samples is smectite abundance (Fig. 18).

6. Subglacial Weathering in the Catchment of Law Glacier

Subglacial chemical weathering processes in the catchment of Law Glacier can be inferred from meteoric ^{10}Be concentrations measured in weathering products in the glacier's sediment deposits at Mt. Achernar moraine. In order for meteoric ^{10}Be to accumulate in weathering products of subglacially-derived sediments at the Mt. Achernar moraine, it must be released from the ice in the catchment area and come into contact with the underlying subglacial material while in meltwater solution. The study area has been ice covered for $> 3 \times 10^7$ years, over three times the applicability of the signal of ^{10}Be with unlikely interglacial exposure (DeConto & Pollard, 2003; Pollard & DeConto, 2009). Therefore, inheritance from the source rock is unlikely.

Measured ^{10}Be concentrations in the samples indicate the relative ratio of meltwater and till in the system. The deposition rates of ^{10}Be in the subglacial environment are calculated to be $\sim 1.35 \times 10^6$ atoms $\text{cm}^{-2} \text{ year}^{-1}$, based on ^{10}Be concentrations in the local ice ($\sim 2 \times 10^5$ atoms g^{-1}) (Raisbeck et al., 1990), the local basal melt rate of $\sim 7.5 \text{ cm year}^{-1}$ (Pattyn, 2010) and ice density of 0.9 g cm^{-3} . Using the total concentrations of $3 - 33 \times 10^7$ atoms g^{-1} in the freshly emerging sediments and assuming a subglacial till density of 2.0 g cm^{-3} (Tulaczyk, 1999), the meltwater input time is calculated to be $\sim 2000 \text{ years cm}^{-1}$ of the till thickness under the ice. We do not have any direct measurements of the till beneath the ice, so it is not possible to reconcile the actual time the sediments were in contact with the meltwater. The calculations above assume an even layer of till that is 1 cm thick. If it were 5 cm, the time in contact with meltwater would be 1.0×10^4 years. We also do not know how far or how fast the water percolates down into the till column, and what ^{10}Be infiltration to depth looks like under the ice.

Meteoric ^{10}Be concentrations of $3 - 32 \times 10^7$ atoms g^{-1} in weathering products and a strong correlation between ^{10}Be concentrations and smectite abundance of the freshly emerging sediments are strong evidence for subglacial chemical weathering at the study site. The accumulation of ^{10}Be in the subglacial till is meltwater dependent, and the strong correlation with smectite clays indicates that the chemical weathering is governed by the input of ice melt and the oxygen therein. Two plausible reactants formed by oxidation reactions in the system could be responsible for the formation of secondary minerals due to dissolution of primary minerals in the source rock. Dissolution could take

place due to carbonic acid, produced by the oxidation of organic matter from the Beacon Supergroup, and due to sulfuric acid, produced by the oxidation of sulfides. Mass balance calculations in Graly et al. (2020) showed that carbonic acid abundance in the system is at least an order magnitude larger than that of sulfuric acid, which is limited by sulfur abundance in the source rock. A likely weathering scenario for the samples starts with the subglacial dissolution of feldspars and pyroxenes by carbonic and/or sulfuric acid as oxygen was introduced to the system with meltwater. Meteoric ^{10}Be in the meltwater would adsorb to the surfaces of fine-grained particles, but preferentially take tetrahedral positions within smectite clay structures as authigenic clay formation took place over hundreds to thousands of years.

The circumstances of the system described here, in which meteoric ^{10}Be is shown to be a useful tracer for subglacial weathering processes, prevail on a larger scale within the continent. Given the widespread nature of the source rocks throughout the TAM (Elliot et al., 2017; Isbell, 1990) and similar basal melting conditions (Pattyn, 2010) across the East Antarctic Ice Sheet, the processes that take place at the Mt. Achnar moraine are likely to be representative of large proportions of the continent. Using the approach presented here, meteoric ^{10}Be could potentially be used to infer subglacial chemical processes on a continent-scale in Antarctica. In order to apply this approach elsewhere in Antarctica, several important conditions need to be met. First, the glaciation history of the location should be considered in order to assess whether ^{10}Be inherited from the source rock is a possibility. The applicability of the signal of ^{10}Be is $\sim 7 \times 10^6$ years, therefore the area should have been ice-covered for at least that long, with no interglacial exposure. Second, the samples, collected from blue ice moraines, should have had minimal exposure on the surface. Therefore, freshly emerging sediments still in contact with/embedded in the ice are preferable. Third, the subglacial geology has to favor the formation of carbonic and/or sulfuric acid, which are responsible for the dissolution of the source rock. Therefore, the source rock composition matters and needs to be considered. The geology of most of East Antarctica is known with less certainty than the geology of the TAM and thus vast areas of the continent might not be as applicable. Sediment at blue ice moraines in the Transantarctic Mountains are good

candidates for this type of study, as the source rock should be relatively similar to the Mt. Achnar moraine system.

7. Conclusions

A sequential chemical extraction procedure was designed and adjusted to optimize the quantification of meteoric ^{10}Be content in weathering products from subglacially-derived sediments at a blue ice moraine in the central Transantarctic Mountains. This study shows that vigorous disaggregation prior to grain size separation and chemical extractions is an important factor for the accurate quantification of ^{10}Be content in the till samples. ^{10}Be bound within the lattice structures of authigenic clays is the dominant fraction of the nuclide in the samples and thus it is important to ensure that fine-grained particles are not unintentionally sieved out during initial sample preparation due to agglomeration to larger grains. For the desorption of adsorbed species, the pH level of the extractant should be maintained above 3.2 to avoid undesired dissolution of more immobile species, and below 3.5 to desorb the maximum amount of ^{10}Be .

Meteoric ^{10}Be concentrations of $2 - 22 \times 10^7$ atoms g^{-1} in abundant weathering products in freshly emerging subglacial sediments strongly indicate chemical weathering in the subglacial environment of the Mt. Achnan moraine catchment area. In the area, which has been ice covered for $> 3 \times 10^7$ years, meteoric ^{10}Be is supplied to the subglacial sediments via subglacial meltwater. The total concentrations of the ^{10}Be in the samples are therefore an indicator for the volume of meltwater that comes into contact with the underlying till. Based on known ^{10}Be concentrations in the ice, basal melt rates from model studies and the ice density, the accumulation time for the concentrations measured in the samples have been calculated to be ~ 2000 years cm^{-1} of till. Although the exact thickness of the till column is unknown, this indicates that the timeframe in which the chemical weathering is taking place in the sediments is on the order of thousands of years. Oxyhydroxides and clays host 64-70% of the total ^{10}Be in the samples, indicating sufficient water and oxygen supply to perform chemical weathering which results in the formation of secondary minerals and amorphous weathering products. Strong correlation of smectite clays with ^{10}Be content ($R = 0.96$) in the samples suggests that authigenic smectite formation takes place in the subglacial environment.

The weathering processes described in this study can potentially be extrapolated to continent-scale weathering rates by examining subglacially derived sediments from across the continent using meteoric ^{10}Be as a tracer. This study introduces an approach

with the potential to estimate Antarctica's contribution to global geochemical cycles via subglacial chemical weathering.

Appendices

Appendix A: Data Tables Corresponding to Graphs and Figures from the Results

Table A2. Fig. 10A data. Solution blank pH levels every two during pH observations (Blank = initial blank pH).

	Blank	2	4	6	8	10	12	14	16
0.05 M	2.96	3.05	3.00	3.03	3.04	3.00	3.07	3.07	3.05
0.11 M	2.82	2.89	2.88	2.88	2.90	2.87	2.93	2.91	3.00
0.22 M	2.64	2.76	2.72	2.75	2.73	2.72	2.79	2.78	2.80
0.44 M	2.52	2.54	2.56	2.54	2.54	2.54	2.61	2.59	2.58

Table A5. Fig. 10B data. Average sample solution pH for each concentration of the acetic acid and their standard deviations. Standard deviations also used to construct error bars on Fig. 10D.

	0	2	4	6	8	10	12	14	16
<i>AVG</i>									
0.05 M	3.46	3.59	3.73	3.69	3.71	3.74	3.69	3.68	3.70
0.11 M	3.18	3.33	3.36	3.39	3.35	3.38	3.36	3.37	3.31
0.22 M	2.93	2.98	3.17	3.13	3.19	3.15	3.15	3.15	3.13
0.44 M	2.73	2.87	2.91	2.95	2.96	2.94	2.93	2.94	2.94
<i>STD DEV</i>									
0.05 M	0.43	0.32	0.40	0.36	0.37	0.40	0.36	0.35	0.36
0.11 M	0.36	0.26	0.27	0.29	0.26	0.29	0.26	0.26	0.21
0.22 M	0.23	0.21	0.24	0.21	0.25	0.23	0.20	0.21	0.19
0.44 M	0.16	0.17	0.18	0.20	0.20	0.20	0.19	0.17	0.17

Table A8. Fig. 10C and 10D data. Sample pH measurements every two hours during pH observations (Blank = initial blank pH before sample addition).

	Blank	0	2	4	6	8	10	12	14	16
<i>0.05 M</i>										
0A	3.01	3.45	3.62	3.66	3.71	3.65	3.67	3.61	3.58	3.62
0B	3.01	3.44	3.65	3.65	3.65	3.62	3.66	3.58	3.56	3.61
0E	2.96		3.52	3.72	3.63	3.70	3.75	3.68	3.71	3.70
1A	3.01	4.01	4.08	4.12	4.12	4.09	4.12	4.03	3.95	4.04
1B	2.96		3.45	3.74	3.64	3.70	3.75	3.69	3.70	3.70
4Q	2.96		3.80	4.21	4.09	4.19	4.24	4.18	4.18	4.18
<i>0.11 M</i>										
0A	2.77	3.19	3.30	3.37	3.35	3.33	3.36	3.34	3.33	3.35
0B	2.77	3.17	3.22	3.31	3.29	3.27	3.29	3.27	3.29	3.31
0E	2.90		3.33	3.34	3.40	3.32	3.42	3.36	3.37	3.18
1A	2.77	3.63	3.61	3.65	3.63	3.59	3.61	3.60	3.57	3.59
1B	2.90		3.33	3.28	3.36	3.30	3.38	3.35	3.35	3.17
4Q	2.90		3.65	3.67	3.79	3.71	3.78	3.73	3.74	3.53
<i>0.22 M</i>										
0A	2.65	2.87	3.06	3.14	3.14	3.15	3.14	3.15	3.11	3.11
0B	2.65	2.93	3.09	3.16	3.14	3.16	3.15	3.15	3.10	3.13
0E	2.57		2.83	3.20	3.10	3.24	3.16	3.14	3.18	3.12
1A	2.65	3.24	3.30	3.38	3.35	3.36	3.35	3.37	3.29	3.30
1B	2.57		2.74	3.12	3.06	3.16	3.09	3.05	3.12	3.06
4Q	2.57		3.10	3.48	3.37	3.52	3.43	3.41	3.46	3.40
<i>0.44 M</i>										
0A	2.47	2.68	2.87	2.83	2.90	2.91	2.88	2.90	2.86	2.89
0B	2.47	2.77	2.93	2.89	2.97	2.99	2.92	2.93	2.92	2.94
0E	2.49		2.88	3.03	3.03	3.07	3.08	3.05	3.07	3.07
1A	2.47	2.93	3.09	3.03	3.10	3.06	3.03	3.04	2.99	3.02
1B	2.49		2.88	2.98	2.97	3.00	3.01	2.98	3.01	3.00
4Q	2.49		2.93	3.07	3.12	3.11	3.12	3.09	3.11	3.10

Table A12. Fig. 11 data. Elemental yields from pH experiments.

Sample	Mg	Ca	Fe	Al	Si	⁹ Be (ppm)	¹⁰ Be ($\times 10^7$ atoms g ⁻¹)	pH at hr 4
<i>0.05 M</i>								
0A	0.01	0.27	0.00	0.00	0.05	0.08	2.27	3.66
0B	0.02	0.29	0.00	0.00	0.05	0.08	4.34	3.65
0E	0.02	0.31	0.00	0.00	0.06	0.11		3.72
1A	0.02	0.75	0.00	0.00	0.04	0.07	0.89	4.12
1B	0.01	0.31	0.00	0.00	0.04	0.07		3.74
<i>0.11 M</i>								
0A	0.02	0.30	0.00	0.00	0.07	0.12	2.77	3.37
0B	0.02	0.37	0.00	0.00	0.09	0.16	7.00	3.31
0E	0.03	0.46	0.00	0.01	0.12	0.20	3.94	3.34
1A	0.03	0.87	0.00	0.00	0.05	0.12	1.67	3.65
1B	0.01	0.43	0.00	0.01	0.08	0.14	0.77	3.28
<i>0.22 M</i>								
0A	0.02	0.50	0.00	0.01	0.16	0.22	5.15	3.14
0B	0.03	0.44	0.00	0.01	0.15	0.20	8.52	3.16
0E	0.03	0.56	0.00	0.01	0.19	0.29		3.20
1A	0.03	0.94	0.00	0.01	0.06	0.19	2.43	3.38
1B	0.02	0.55	0.00	0.02	0.15	0.23		3.12
<i>0.44 M</i>								
0A	0.03	0.64	0.00	0.02	0.23	0.31	6.56	2.83
0B	0.04	0.59	0.00	0.01	0.22	0.28	11.72	2.89
0E	0.04	0.62	0.00	0.02	0.23	0.35		3.03
1A	0.03	1.01	0.00	0.02	0.12	0.26	3.16	3.03
1B	0.02	0.59	0.00	0.03	0.19	0.25		2.98

Table A11. Fig. 12 data. ¹⁰Be yield from methods 1 and 2.

Sample (method)	Adsorbed	Oxyhydroxides	Residual	Clay lattices	¹⁰ Be (10^7 atoms g ⁻¹)
0B (1)	6.38	13.42	1.58	1.00	22.39
0B (2)	6.46	10.85	2.53	2.32	22.17
0E (1)	2.70	7.10	1.04	1.09	11.93
0E (2)	2.69	4.63	1.55	0.52	9.39
1B (1)	0.56	0.96	0.28	0.14	1.94
1B (2)	0.50	0.75	0.33	0.22	1.79

Table A6. Fig. 14 data. Measured Percent fractionation of ^{10}Be concentrations in all samples from all methods.

Sample (method)	Adsorbed	Oxyhydroxides	Residual	Clay lattices
1B (1)	0.29	0.50	0.15	0.07
0E (1)	0.23	0.60	0.09	0.09
0B (1)	0.29	0.60	0.07	0.04
1B (2)	0.28	0.42	0.19	0.12
0E (2)	0.29	0.49	0.16	0.06
0B (2)	0.29	0.49	0.11	0.10
0B (3)	0.32	0.22	0.02	0.45
1A (3)	0.27	0.34	0.05	0.35
0A (3)	0.35	0.04	0.02	0.60
4Q (3)	0.17	0.35	0.04	0.44

Table A14. Fig. 14 and Fig. 15 data. Measured ^{10}Be concentrations in all samples from all methods.

Sample (method)	Adsorbed	Oxyhydroxides	Residual	Clay lattices	Total ^{10}Be
1B (1)	5.61E+06	9.64E+06	2.83E+06	1.35E+06	1.94E+07
1B (2)	4.97E+06	7.46E+06	3.33E+06	2.19E+06	1.79E+07
0E (1)	2.70E+07	7.10E+07	1.04E+07	1.09E+07	1.19E+08
0E (2)	2.69E+07	4.63E+07	1.55E+07	5.22E+06	9.39E+07
0B (1)	6.38E+07	1.34E+08	1.58E+07	9.98E+06	2.24E+08
0B (2)	6.46E+07	1.09E+08	2.53E+07	2.32E+07	2.22E+08
0B (3)	1.04E+08	7.06E+07	6.27E+06	1.45E+08	3.26E+08
1A (3)	2.60E+07	3.33E+07	4.61E+06	3.42E+07	9.82E+07
0A (3)	4.91E+07	5.35E+06	2.18E+06	8.47E+07	1.41E+08
4Q (3)	2.28E+08	4.72E+08	4.75E+07	5.85E+08	1.33E+09

Table A15. Calculations for the normalization to method 3, using relative differences of sample 0B.

	Adsorbed	Oxyhydroxides	Residual	Clay lattices	Total ^{10}Be ($\times 10^7$ atoms g^{-1})
0B (1)	6.38	13.42	1.58	1.00	22.39
0B (2)	6.46	10.85	2.53	2.32	22.17
AVG M1 & M2	6.42	12.14	2.06	1.66	22.28
0B (3)	10.40	7.06	0.63	14.53	32.61
Difference	1.62	0.58	0.30	8.77	1.46

Table A9. Fig. 16 data. Fractionation of ^{10}Be in all samples. Samples 0E and 1B normalized to method 3.

	Adsorbed	Oxyhydroxides	Residual	Clay lattices	Total ^{10}Be ($\times 10^7$ atoms g^{-1})
0A	4.91	0.53	0.22	8.47	14.13
0B	10.40	7.06	0.63	14.53	32.61
0E	4.36	3.41	0.39	7.05	15.21
1A	2.60	3.33	0.46	3.42	9.82
1B	0.86	0.50	0.09	1.55	3.00
4Q	22.75	47.20	4.75	58.51	133.22

Table A10. Fig. 17 data. Fractionation of ^{10}Be (in %) between grain sizes and phases.

Grain Fraction	Adsorbed	Oxyhydroxides	Residual	Clay lattices	Total
<i>0A</i>					
FS	3.00%	1.33%	0.38%		4.71%
St	6.48%	4.12%	1.10%		11.70%
CC	7.36%			15.21%	22.56%
FC	10.95%			50.07%	61.03%
<i>Total</i>	27.79%	5.45%	1.48%	65.28%	100.00%
			<i>Clay + OOH:</i>	70.73%	
<i>0B</i>					
Ms	2.42%	1.40%	0.30%		4.11%
FS	3.69%	1.17%	0.40%		5.26%
St	9.29%	8.46%	2.02%		19.77%
CC	10.25%			31.34%	41.58%
FC	13.90%			15.38%	29.28%
<i>Total</i>	39.54%	11.03%	2.71%	46.71%	100.00%
			<i>Clay + OOH:</i>	57.74%	
<i>1A</i>					
FS	4.79%	4.10%	1.21%		10.10%
St	11.44%	20.14%	4.96%		36.54%
CC	7.06%			18.30%	25.36%
FC	9.49%			18.51%	28.00%
<i>Total</i>	32.78%	24.24%	6.18%	36.81%	100.00%
			<i>Clay + OOH:</i>	61.05%	
<i>4Q</i>					
FS	2.85%	6.99%	1.43%		11.27%
St	8.91%	16.18%	4.01%		29.09%
CC	7.15%			43.74%	50.89%
FC	3.14%			5.61%	8.75%
<i>Total</i>	22.04%	23.17%	5.44%	49.35%	100.00%
			<i>Clay + OOH:</i>	72.52%	

Table A11. Fig. 17 data. Fractionation of ^{10}Be between grain sizes and grain phases.

Grain Fraction	Adsorbed	Oxyhydroxides	Residual	Clay lattices
<i>0A</i>				
FS	3.89E+06	1.73E+06	4.92E+05	
St	8.41E+06	5.35E+06	1.43E+06	
CC	9.54E+06			1.97E+07
FC	1.42E+07			6.50E+07
<i>0B</i>				
MS	3.85E+06	2.23E+06	4.73E+05	
FS	5.88E+06	1.86E+06	6.36E+05	
St	1.48E+07	1.35E+07	3.21E+06	
CC	1.63E+07			4.99E+07
FC	2.21E+07			2.45E+07
<i>1A</i>				
FS	2.90E+06	2.48E+06	7.35E+05	
St	6.93E+06	1.22E+07	3.01E+06	
CC	4.28E+06			1.11E+07
FC	5.75E+06			1.12E+07
<i>4Q</i>				
FS	2.16E+07	5.30E+07	1.08E+07	
St	6.75E+07	1.22E+08	3.04E+07	
CC	5.41E+07			3.31E+08
FC	2.38E+07			4.25E+07

Table A12. Fig. 18 data. Clay abundances in the samples in weight%

	Illite	Chlorite	Kaolinite	Smectite	Total ^{10}Be ($\times 10^7$ atoms g^{-1})
0A	14.319	0.301	0.257	12.871	14.1
0B	14.7	0.8	2.4	20	32.6
0E	13.9	0.2	0.5	15	15.6
1A	14.7	3.6	2.2	10.2	9.8
1B	16.8	0.4	0.6	8.7	2.7
4Q	14.7	0.6	1.4	6.7	133.2

Table A13. Table 5 data. Summary of correlation coefficients and significance of correlation for total ^{10}Be concentrations and elemental extracts in each extraction, including sample 4Q and excluding sample 4Q.

Acetic Acid Extracts

With 4Q	Mg	Ca	Na	K	Fe	Al	Si	^9Be
P-value	0.004	0.016	0.005	0.008	0.018	0.009	0.628	0.653
R	0.948	0.893	0.943	0.925	0.887	0.920	0.253	0.235
R ²	0.899	0.798	0.890	0.856	0.786	0.847	0.064	0.055
Without 4Q	Mg	Ca	Na	K	Fe	Al	Si	^9Be
P-value	0.594	0.969	0.394	0.914	0.973	0.913	0.726	0.894
R	0.325	0.024	0.497	0.067	0.021	0.068	0.217	0.083
R ²	0.105	0.001	0.247	0.005	0.000	0.005	0.047	0.007

HCl Extracts

With 4Q	Mg	Ca	Na	K	Fe	Al	Si	^9Be
P-value	0.002	0.796	0.011	0.244	0.070	0.499	0.381	0.376
R	0.962	0.137	0.913	0.564	0.776	0.348	0.441	0.446
R ²	0.925	0.019	0.834	0.318	0.602	0.121	0.195	0.199
Without 4Q	Mg	Ca	Na	K	Fe	Al	Si	^9Be
P-value	0.223	0.647	0.838	0.081	0.662	0.384	0.970	0.877
R	0.663	0.281	0.128	0.832	0.269	0.506	0.024	0.096
R ²	0.439	0.079	0.016	0.691	0.072	0.256	0.001	0.009

Totally Digested Residual Particles

With 4Q	Mg	Ca	Na	K	Fe	Al	Si	^9Be
P-value	0.796	0.431	0.957	0.983	0.510	0.767	n/a	0.005
R	0.137	0.401	0.029	0.012	0.340	0.157	n/a	0.942
R ²	0.019	0.160	0.001	0.000	0.116	0.025	n/a	0.887
Without 4Q	Mg	Ca	Na	K	Fe	Al	Si	^9Be
P-value	0.574	0.820	0.997	0.837	0.177	0.743	n/a	0.722
R	0.341	0.142	0.002	0.129	0.712	0.203	n/a	0.220
R ²	0.116	0.020	0.000	0.017	0.507	0.041	n/a	0.049

Totally Digested Clay Particles

With 4Q	Mg	Ca	Na	K	Fe	Al	Si	^9Be
P-value	0.127	0.021	0.034	0.268	0.168	0.440	n/a	0.428
R	0.692	0.881	0.846	0.541	0.643	0.393	n/a	0.403
R ²	0.479	0.775	0.715	0.292	0.414	0.155	n/a	0.163
Without 4Q	Mg	Ca	Na	K	Fe	Al	Si	^9Be
P-value	0.411	0.212	0.805	0.261	0.777	0.876	n/a	0.880
R	0.482	0.674	0.154	0.623	0.176	0.098	n/a	0.094
R ²	0.233	0.454	0.024	0.389	0.031	0.010	n/a	0.009

Table A14. Fig. 19 data. Elemental yields of all samples from all methods.

Sample (Method)	Mg	Ca	Na	K	Fe	Al	Si	9Be (ppm)
<i>Acetic Acid Extracts</i>								
0B (1)	0.03	0.41	0.02	0.01	0.00	0.00	0.12	1.32
0E (1)	0.03	0.41	0.01	0.01	0.00	0.01	0.12	1.90
1B (1)	0.01	0.45	0.01	0.01	0.00	0.01	0.10	1.72
0B (2)	0.03	0.42	0.02	0.01	0.00	0.00	0.12	1.75
0E (2)	0.03	0.43	0.01	0.01	0.00	0.00	0.13	2.26
1B (2)	0.01	0.46	0.01	0.01	0.00	0.01	0.10	1.94
0A (3)	0.08	1.68	0.02	0.04	0.01	0.03	0.50	0.57
0B (3)	0.10	1.77	0.03	0.04	0.01	0.02	0.54	0.64
1A (3)	0.10	2.76	0.02	0.07	0.01	0.03	0.59	0.90
4Q (3)	1.29	8.00	10.09	0.32	0.03	0.14	0.18	0.17
<i>HCl Extracts</i>								
0B (1)	0.15	0.61	0.01	0.05	0.38	0.64	0.59	2.22
0E (1)	0.15	0.56	0.01	0.06	0.47	0.76	0.66	2.01
1B (1)	0.13	0.48	0.01	0.05	0.44	0.62	0.63	1.76
0B (2)	0.13	0.49	0.01	0.04	0.33	0.56	0.51	2.28
0E (2)	0.12	0.45	0.01	0.05	0.37	0.61	0.54	2.26
1B (2)	0.11	0.37	0.01	0.04	0.35	0.50	0.51	1.71
0A (3)	0.08	0.25	0.01	0.03	0.58	0.52	0.54	-
0B (3)	0.11	0.42	0.01	0.04	0.42	0.64	0.65	0.32
1A (3)	0.12	0.77	0.00	0.05	0.69	0.77	0.94	0.45
4Q (3)	0.24	0.50	0.03	0.05	0.93	0.69	0.84	0.41
<i>Totally Digested Residual Particles</i>								
0B (1)	0.10	0.47	-	1.41	2.01	2.62	-	3.91
0E (1)	0.00	0.07	-	1.62	1.46	2.49	-	2.19
1B (1)	-	0.23	-	1.36	2.51	2.03	-	4.16
0B (2)	0.08	0.40	-	1.45	1.99	2.88	-	4.48
0E (2)	0.08	0.43	-	1.78	2.71	2.95	-	5.18
1B (2)	0.00	0.60	-	2.01	3.65	3.05	-	7.41
0A (3)	0.01	0.03	0.97	2.37	2.87	4.72	-	-
0B (3)	0.91	0.27	0.99	2.36	3.04	7.04	-	-
1A (3)	0.00	0.02	0.68	1.68	2.56	4.11	-	0.23
4Q (3)	0.04	0.04	0.56	1.96	3.13	4.78	-	0.66
<i>Totally Digested Clay Particles</i>								
0B (1)	0.06	0.19	-	2.08	7.38	5.37	-	8.88
0E (1)	0.74	0.85	-	1.37	7.53	3.03	-	16.18
1B (1)	0.22	0.30	-	2.84	8.95	4.80	-	7.68
0B (2)	1.67	1.53	-	2.20	11.87	5.56	-	26.92
0E (2)	0.13	0.51	-	5.05	16.06	10.01	-	40.47
1B (2)	0.37	1.28	-	2.89	10.57	5.41	-	11.49
0A (3)	0.57	0.36	0.53	2.44	8.15	13.82	-	3.07
0B (3)	0.13	0.11	0.34	1.75	8.26	9.89	-	2.60
1A (3)	2.25	0.60	0.58	4.89	9.32	20.29	-	4.10
4Q (3)	3.06	2.29	1.27	5.27	12.51	17.38	-	0.00

Table A15. Sample mass, spike mass and ^{10}Be data from PRIME Lab.

Sample*	Sample Mass (g)	^9Be Spike (μg)	$^{10}\text{Be}/^9\text{Be}$ (10^{-15})	Uncertainty (10^{-15})	^{10}Be (10^7 atoms g^{-1})
<i>Method 1</i>					
0B HCl	1.007	263.165	2783.050	38.351	13.425
0B Res TD	1.007	274.928	369.600	8.023	1.583
0B Clay TD	1.007	267.121	238.521	6.361	0.998
0E AA	1.009	265.143	575.855	15.587	2.701
0E HCl	1.009	265.039	1274.860	16.322	7.098
0E Res TD	1.009	265.767	337.619	7.526	1.042
0E Clay TD	1.009	215.175	313.008	7.519	1.085
1B AA	1.004	291.792	98.735	5.166	0.561
1B HCl	1.004	263.061	199.240	5.884	0.964
1B Res TD	1.004	265.351	61.720	2.122	0.283
1B Clay TD	1.004	259.938	34.414	1.534	0.135
1B AA	1.007	245.676	1585.410	29.799	6.384
<i>Method 2</i>					
0B AA	1.002	262.124	1424.400	35.299	6.461
0B Clay HCl	1.002	264.206	2149.040	26.098	10.855
0B Res TD	1.002	185.923	950.604	15.706	2.534
0B Clay TD	1.002	271.389	573.905	10.899	2.316
0E AA	1.006	264.830	611.056	12.916	2.691
0E Clay HCl	1.006	262.228	994.993	15.345	4.628
0E Res TD	1.006	258.897	434.474	8.912	1.548
0E Clay TD	1.006	268.682	133.176	4.546	0.522
1B AA	1.003	263.373	96.661	5.634	0.497
1B Clay HCl	1.003	244.010	166.155	3.968	0.746
1B Res TD	1.003	266.288	74.859	2.305	0.333
1B Clay TD	1.003	244.843	61.106	2.242	0.219
<i>Method 3</i>					
0A FS AA	1.020	277.635	222.223	9.918	1.128
0A ST AA	0.789	276.281	483.176	13.411	3.155
0A CC AA	0.431	273.887	567.742	14.797	6.548
0A FC AA	0.719	275.449	780.607	13.050	5.851
0A FS HCl	1.020	275.240	99.651	7.155	0.501
0A ST HCl	0.789	275.345	300.567	11.216	2.006
0A FS TD	1.020	268.578	34.347	3.034	0.143
0A ST TD	0.789	280.966	101.867	5.459	0.536
0A CC TD	0.431	270.972	1523.810	21.881	8.870
0A FC TD	0.719	270.764	2607.320	52.969	17.524
0B MS AA	1.007	275.553	260.922	7.257	1.297
0B FS AA	0.647	280.237	408.925	6.887	3.079
0B ST AA	0.648	278.259	993.156	16.630	7.750
0B CC AA	0.476	277.843	1041.930	25.163	11.637
0B FC AA	0.614	277.531	1451.350	18.337	12.226

0B MS HCl	1.007	272.846	144.801	4.352	0.750
0B FS HCl	0.647	259.834	127.233	4.743	0.976
0B ST HCl	0.648	274.095	894.531	12.824	7.057
0B MS TD	1.007	273.262	36.133	1.896	0.159
0B FS TD	0.647	278.780	83.501	3.647	0.333
0B ST TD	0.648	275.657	417.151	9.316	1.683
0B CC TD	0.476	278.467	3560.260	33.726	35.592
0B FC TD	0.614	280.550	1734.390	29.696	13.528
1A FS AA	1.002	279.509	174.937	5.316	0.833
1A ST AA	1.054	265.143	384.198	8.379	1.891
1A CC AA	0.471	184.257	358.109	9.489	2.610
1A FC AA	0.348	275.136	330.844	12.572	4.749
1A FS HCl	1.002	275.136	135.694	5.952	0.712
1A ST HCl	1.054	277.947	660.296	13.508	3.329
1A FS TD	1.002	253.796	49.030	3.810	0.211
1A ST TD	1.054	272.638	211.423	8.073	0.820
1A CC TD	0.471	268.370	634.029	13.656	6.764
1A FC TD	0.348	269.307	1292.560	26.814	9.264
4Q FS AA	1.005	266.808	1521.570	33.053	5.966
4Q ST AA	0.722	276.281	4526.270	93.000	25.990
4Q CC AA	0.779	260.666	5643.890	63.865	19.315
4Q FC AA	0.275	260.146	2478.300	41.007	23.996
4Q FS HCl	1.005	275.553	2595.220	47.004	14.653
4Q ST HCl	0.722	279.092	6382.860	124.127	47.195
4Q FS TD	1.005	266.808	763.884	19.202	2.995
4Q St TD	0.722	276.281	2036.930	45.557	11.696
4Q CC TD	0.779	260.562	52727.896	516.124	118.252
4Q FC TD	0.275	260.458	6545.491	176.467	42.901
<i>Acetic Acid pH Experiments</i>					
0A 0.05 M	1.055	258.272	511.574	11.739	2.273
0B 0.05 M	1.026	257.856	922.404	38.626	4.343
1A 0.05 M	1.013	258.272	191.271	7.953	0.889
0A 0.11 M	1.383	260.770	809.018	19.349	2.772
0B 0.11 M	1.028	259.417	1526.230	26.118	6.999
1A 0.11 M	1.026	258.584	364.035	11.940	1.675
0A 0.22 M	1.018	260.979	1078.600	24.585	5.153
0B 0.22 M	1.117	259.417	2022.830	44.931	8.521
1A 0.22 M	1.017	260.250	524.696	12.721	2.431
0A 0.44 M	1.077	259.834	1435.400	30.020	6.565
0B 0.44 M	1.078	260.146	2639.760	99.557	11.720
1A 0.44M	1.040	259.313	696.062	18.429	3.165
<i>High pH Na₂CO₃ Leach</i>					
0A Na	1.383	259.625	7.470	1.814	0.022
0B Na	1.028	260.354	6.681	1.426	0.026
1A Na	1.026	258.897	5.807	2.035	0.023

*Terms indicate size fractions and acid extractions. Res = Residual fraction (non-clay silicates), MS = Medium Sand, FS = Fine Sand, St = Silt, CC = Coarse Clay, FC = Fine Clay. AA = Acetic Acid, HCl = Hydrochloric Acid, TD = Totally Digested.

Appendix B: Step-by-Step Laboratory Procedures

Blend + Sonication

1. Weigh ~5g of each sample
 - a. Make a judgment call:
 - i. Hand-pick out large ($>$ sand, 2mm) particles, or
 - ii. Sieve through 2mm mesh
2. 400-600 ml DI water in blender
3. Sample poured in
4. Blend for 60 seconds
5. Transfer to [plastic] beaker
6. Sonicate for 10 minutes
7. Repeat steps 3-5 once

Wet sieving

1. Out of the settled sediments from the initial 10 min settling, separate medium sand ($>250\text{ }\mu\text{m}$) from fine sand ($> 63\text{ }\mu\text{m}$) and silt ($<63\text{ }\mu\text{m}$)
2. Pour sample over small sieve of the right mesh size (250/63 μm)
3. Over a ceramic/collecting tray bowl, use squirt bottle to wash sample through sieve with DI water trying to use as little water as possible
4. Transfer sieved material into falcon tubes and combine into one by centrifuging
5. Final products:
 - i. 1 tube with medium sand ($>250\text{ }\mu\text{m}$)
 - ii. 1 tube with fine sand ($>63\text{--}250\text{ }\mu\text{m}$)

Stoke's Gravity Settling:

Note: first calculate the settling time based on Stoke's law

1. Begin with 10 min settling to separate out sand and silt
 - a. Decant suspended material (clays) after 10 mins
2. In a 1L beaker, settle for 1 hr to separate silt from clay
 - a. Decant into new beaker
 - b. Residue into labeled falcon tube
1. In a 1L beaker, settle for [*the calculated amount of*] hrs to separate coarse clays from fine clays
 - a. Decant fine clays into new beaker
 - b. Residue into labeled falcon tube
2. Centrifuge fine clays, decant water and transfer into falcon labeled tubes
3. Final products:
 - a. 1 tube w/sand --> Sieve w. 250 and 63 μm sieves
 - b. 1 tube w/silt
 - c. 1 tube w/coarse clay ($>0.5\text{ }\mu\text{m}$)
 - d. 1 tube with fine clay ($<0.5\text{ }\mu\text{m}$)

Acetic Acid Extractions

1. Label new tubes *clearly and descriptively*
 - a. Weigh tubes
 - b. Pour supernatant into new tubes
2. Label and weigh 50 ml plastic tubes
3. Put sample (ca. 1 g) in tube
 - a. Record sample weight
4. Determine the volume required to cover all samples + one blank + 3 extra tubes (just in case)
5. Mix acid of desired molarity and volume
6. Pour 40 ml into one empty falcon tube labeled '*acid blank*', using repeat pipette
7. Pour 40 ml into tubes with samples, using repeat pipette
8. Vortex to make sure no sediment is stuck to the bottom and make sure lids are secure
 - a. Leave on vibrating table for 16 hrs
 - b. Centrifuge for 15 mins & decant leachate into a fresh, labeled tube
9. 20 ml DI water into sample tubes
 - a. Vibrating table for 2 hours
 - b. Centrifuge 15 mins
 - c. Dispose of water

HCl Extractions

1. Label new tubes *clearly and descriptively*
 - a. Weigh tubes
 - b. Pour supernatant into new tubes
2. Determine the volume required to cover all samples + one blank + 3 extra tubes
3. Mix acid of desired molarity and volume
4. Pour 40 ml into one empty plastic tube labeled '*acid blank*'
5. Pour 40 ml into tubes with samples - use empty styrofoam tray to elevate the samples
6. to make pipetting easier
 - a. Vortex to make sure no sediment is stuck to the bottom
7. Make sure the lids are secure
 - a. Leave on vibrating table for 24 hrs
 - b. Centrifuge for 15 mins & decant leachate into a fresh, labeled tube
8. 20 ml DI water into sample tubes
 - a. Vibrating table for 2 hours
 - b. Centrifuge 15 mins
 - c. Dispose of water

pH Experiments – Acetic Acid

1. pH meter turned on and let warm up for at least 30 minutes, then calibrated.
2. 50 ml plastic tubes labeled and weighed
3. 1 g of each sample (<63 mm) weighed and placed in tubes
4. Acetic acid prepared at following concentrations:
 - a. a: 0.05 M
 - b. b: 0.11 M
 - c. c: 0.22 M
 - d. d: 0.44 M
5. pH in each beaker measured with a pH meter
6. 40 ml of acid poured into pertinent tubes using repeat pipette. Note time, this is the start of the 16-hour leach.
7. Tubes closed and vortexed for ca. 5 seconds, making sure the whole sample is in suspension
8. pH in each tube measured and recorded.
9. All tubes placed on vibrating table, making sure lids are securely closed.
10. pH recorded every 2 hours. Since measuring the pH of 8 tubes took 20-40 minutes, only half of the tubes were taken off the vibrating table at a time in this case, to maximize time of each sample on vibrating table.
11. After pH had been recorded after 16 hours, samples were centrifuged for 15 minutes and acids decanted into new 50 ml falcon tubes (labeled: xxxx v AA).
12. 45 ml of DI water added to sample tubes and placed on vibrating table for 2 hours to wash. Centrifuged for 15 minutes, water carefully decanted into sink.

Experimental Sodium Carbonate Extraction

1. The heated sonicator turned on 3 hours prior to allow water to reach 60°C.
2. 200 ml 2M sodium carbonate solution prepared in a glass beaker.
 - a. 43.3427 g of Na_2CO_3 weighed and slowly poured into a glass beaker with 200 ml DI water on hot plate with stirrer on.
 - b. Beaker removed from hot plate once all powder was dissolved
 - c. pH recorded in beaker
3. 40 ml of base poured into samples+ blank tube using
 - a. repeat pipette (these are the 0.11M AA samples from 16.MAR 2020).
4. All tubes placed in a plastic tube rack in sonicator and temperature checked every 15 mins (making sure it stays between 55 and 65°C) for 5 hours.
5. Tubes with samples shaken every 15 minutes to make sure samples stay in suspension/do not stick to the bottom of the tube.
6. After 5 hours, centrifuged for 15 minutes and decanted into new 50 ml
 - a. falcon tubes (labeled: xxxx b Na).
7. 45 ml water added to sample tubes and placed on vibrating table for washing. Centrifuged and water decanted into sink.

ICP-OES Analysis

Setting up a run:

The ICP-OES needs to run “hot”, i.e. several hours of pumping than at least half of hour of running with the torch lit are needed for best performance. I always start up the instrument first and then make my standards while it is pumping etc.

1. Open the N2 and make sure that the gauge is 275-825kPa coming out of the tank and that sufficient N2 is in the tank.
2. Open winlab software
3. Set ICP to high purge.
4. Let run for at least 2 hours. In the mean time you can make standards.
5. Turn on the water chiller (pressure should be set between 200-550 kPa, flow rate set at 4 L/min, and temperature set between 15-25 °C)
6. Open the Ar and make sure that the gauge is 550-825 kPa coming out of the tank and that you have sufficient Ar in the tank.
7. Hook up the tubing around the peristaltic pump. Make sure it clicks into place and the red and black go the correct directions.
8. Switch the source from H2O to nitric acid tanks.
9. Turn on the Plasma. The plasma must run for at least half an hour (though closer to an hour is probably preferable). In the meantime, dilute the samples.

Making Standards:

It is best practice to make a separate set of standards for each extraction you wish to analyze. If this is not practical, make the standards out of the strongest extractant.

1. Determine the dilution to be employed. The rest of the SOP assumes a 1:10 dilution. Adjust the calculations accordingly for a different dilution
2. Select elements to be analysed. I typically use Na, Mg, Ca, K, Fe, Al, Si as major elements. Minor and trace elements of potential interest depending on the application have included Ti, Mn, P, B, S, Be.
3. Select element volumes. I typically used 100 µl for major elements and 10 µl for minor or trace elements unless there is some reason to suspect that the extraction will be particularly enriched an element. For instance you could use 1000 µl Ca for an acetic acid extraction or 1000 µl Fe for HCl extraction (as those extractions tend to favour those respective elements).
4. Set a 50 ml falcon tube on scale and zero out. Add each of the elements in the desired volume, recording the weight and re-zeroing between each element. Use a fresh pipette tip every time.
5. Add 4 ml of extraction blank to the tube.
6. Sum the total weight of all elements. Add 40 ml minus this sum of mille-q water to tube. Weigh and record. Cap tube and shake to homogenize. You have made standard 4.
7. Place a new 50 ml tube on the scale and re-zero.
8. Add 4 ml of extraction blank to the tube. Add 40 ml of mille-q water. Cap and shake to homogenize. This is standard 1 (aka calib blank).

9. Rezero another tube. Add 10 ml of standard 1 and 10 ml of standard 4. Weigh the actual mass of each. Do not cross contaminate with pipette tips. Cap and shake to homogenize. This is standard 3.
10. Follow that same procedure but with 15 ml of standard 1 and 5 ml of standard 4. This is standard 2.
11. For each standard pour off 10 ml into a 15 ml falcon tube. These can be used as check standards and blanks. Standard 4 will have 30 ml of material, so 2 check standards can be made. Depending on the number of elements you are running and number of check standards you are using, you may need more material, so judge accordingly.

Setting up Samples:

1. Organize all of the samples you intend to run in rows that match the order in which you will place samples on the ICP.
2. Intersperse premade blanks and check standards throughout. In general, there should be blank or a check standard every 4-5 samples. For example: blank – 4 unknowns – check standard – 4 unknowns – blank – 4 unknowns – check standard, etc. There should also be a blank and check standard whenever you switch to a new extraction. i.e. If you go from an acetic leach to an HCl leach.
3. For all of the unknowns pipette in 1 ml of sample. If you need to make more blanks, add these as well. Add 10 ml of milliq water to all samples that need dilution.
4. Place the samples in the racks making sure the racks are properly emplaced so the auto sampler will reach them.

Creating a method:

1. On the home screen, click <File>, <New>, <Method>. Alternatively you can edit an existing method. (<File>, <Open>, <Method>.)
2. In 'Create New Method' window, click <OK> (to selecting aqueous solution).
3. Next window will say 'Method Editor'. On the 'define elements' tab, enter the elemental abbreviations being analyzed. The software will choose the most optimal wavelengths for each element. This can be changed if an element needs to be analyzed at a different wavelength.
4. On the same window, 'Settings' tab, make sure 'replicates' reads '3 ', unless, triplicates are not needed. The software always defaults to 1 reading unless specified.
5. In the 'Method Editor', click <Calibration> tab (bottom).
6. On the 'Define Standards' tab (right), enter the autosampler (A/S) locations of each standard. If your 'SI/Standard 1' is a blank, enter it in the 'Calib Blank' section. All other non-blank calibration standards should be entered in the 'Calib Std' section.
7. At this point you will need to perform mass calculations for each of the standards that included in standard 4. These are calculated by the added volume * the concentration (usually 1000) / total volume (44 ml). Set all of the standard 1 values to zero. Use the weighed masses from standards 2 and 3 to weight the proportion of standard 4 values in the other two standards.

Creating a sample information file:

1. On the home page toolbar, click <File>, <New>, <Sample Info File>.
2. In the 'Sample Information File Editor' window, enter the A/S location and a briefID for each sample. Remember, the A/S locations and the sample numbers are NOT the same identifiers.
3. DO NOT enter the sample weights at this time

Calibration:

1. On the home page toolbar, click the <Autosampler> icon.
2. The 'Automated Analysis Control' window will appear. On the 'Set Up' tab, insure the selected method reads in the 'No. 1' row. The selected information file should also appear in the correlating window.
3. Double click on <Open> to the left of the 'Results Data Set Name' window. Enter a specific name for the data. Again, include your initials, date, and what the samples are.
4. Next, click the <Analyze> tab (bottom of the 'Automated Analysis Control' window.
5. Click <Rebuild List>.
6. If the calibration standards are in the correct A/S locations, click the <Calibrate> icon.
7. The autosampler will now begin to analyze the first calibration standard.
8. On the homepage toolbar, clicking the <Spectra>, <Results>, and <Calibration> icon, all allow you to see data as the instrument acquires it.
9. RSD values in the results summary should be around 1%. These values will always be higher for blanks.
10. Correlation coefficients should be AT LEAST 0.9999x for all elements used in the calibration.

Analyzing the Samples

1. On the 'Automated Analysis Control' window, click <Rebuild List>. The sample ID's should now be listed after the calibration standards.
2. Click <Analyze Samples>. The autosampler will now go to your first sample. If the autosampler doesn't move right away, it is either rinsing or waiting for the spectrophotometer initialization process to finish.

Finishing the Run

1. When the run is completed, rinse the autosampler for 5 minutes with MQ <Analysis>.
2. <Autosampler>, <Go to location 5>. Make sure the blue cap is off of the centrifuge tube labeled 'MQ' in A/S location 5.
3. Then send it to an empty position for 5 minutes (this allows the lines and nebulizer chamber drain and empty). <Analysis>, <Autosampler>, <Go to location x> (x = any A/S location that doesn't have a centrifuge tube in it).
4. When the drainage is finished, send the autosampler back to the 'wash' position: Click <Analysis>, <Autosampler>, <Go to Wash>

5. Turn off the main peristaltic pump. Click <System>, <Pump On/Off>
6. Completely release the tubing on the main pump (the wheel).
7. Turn off plasma. Click the <Plasma> icon, then the 'Off' switch.
8. Turn off the N₂ and Ar gases.
9. Relocate the autosampler arm to the wash position and then raise the arm up.
<Analysis>,
10. <Autosampler>, <Go to wash>, <A/S Probe Up/Down>.
11. Press the power button on the water chiller.
12. Check the waste carboy level (if full, waste needs to be neutralized before discarding).

XRD Clay Mount Preparation

1. Turn heating plate on (2-3)
2. Agitate samples so that particles are suspended in water
 - a. Add a few ml of DI water to samples if needed,
 - b. Shake and/or vortex so that nothing's sticking to the bottom of the tube
3. Make sure slide is clean, rinse with DI if needed
4. Place slide on plate
5. Use 100 μ L repeat pipette/disposable pipette to extract clays from tube and place on slide
 - a. Make sure to use a fresh tip/disp. pipette for each sample
6. Label samples on slides/make notes to know which sample is which on the slide
7. Let samples dry

Preparation for AMS Analysis: ⁹Be spike

1. Label 15 ml plastic tube for each sample & blank to spike:
2. Record the volume of liquid in sample tube *before spike*
3. Weigh 15 ml tube
4. Add 14.3 ml of sample (3 x 4.7 ml) using repeat pipette
5. Record weight of tube + sample
6. Add 250 μ l of Be spike using a repeat pipette, record the weight
7. Record volume of sample in sample tubes after extracting sample for spike
8. Make sure the lids are screwed on tightly
9. Seal with parafilm
10. Put together a list of all tubes w/sample name and chemicals in tube to print and send with samples to Purdue

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