10Be Chronology of Moraines Deposited During the Last Glaciation by the Khumbu Glacier, Nepalese Himalaya

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10Be CHRONOLOGY OF MORAINES DEPOSITED DURING THE LAST GLACIATION BY THE KHUMBU GLACIER, NEPALESE HIMALAYA

By
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B.S. University of Maine, 2019

A THESIS
Submitted in Partial Fulfillment of the Requirements for the Degree of Master of Science (in Earth and Climate Sciences)

The Graduate School
The University of Maine
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Understanding the global pattern of glaciation can aid interpretation of what caused late-Pleistocene glacial cycles. Here I investigate the glacial history of the Khumbu Glacier of the Mount Everest region of Nepal. Prominent hypotheses for Himalayan glaciation suggest important roles for orbitally modulated insolation change and the Asian summer monsoon. Here I test these hypotheses by developing a $^{10}$Be surface-exposure chronology of moraines constructed by the Khumbu Glacier during the last glacial period. My chronology is underpinned by detailed glacial-geomorphic mapping constructed by use of drone and satellite imagery. The ages presented in this study indicate that the Khumbu glacier stood at maximum positions within the Dingboche moraine complex at ~67.1 ka, 38.1 ka, 36.7 ka, 29.7 ka, ~22.6 ka, 17.8 ka. The data indicate ice-surface lowering of ~100 m from the top of the Pheriche moraine to the Pheriche recessional landforms between 17.8 ka and 15.8 ka. These data suggest that moraine construction occurred throughout an entire orbital insolation cycle. Therefore, insolation intensity cannot explain the signature of Khumbu glacier fluctuations during the last glaciation.
Likewise, peak glaciation occurred during a period of relatively weak monsoon intensity, suggesting that monsoonal forcing mechanisms (i.e., precipitation and/or cloudiness) are insufficient for explaining the pattern of glaciation indicated by the chronology presented here. The Khumbu Glacier chronology therefore raises a key question: If insolation and local monsoonal forcing did not drive the last glacial cycle in the high Himalaya, then what climatic factor(s) did? In this regard, I pose a preliminary suggestion that tropical heat fluxes inferred from Indian Ocean sea-surface temperatures, perhaps coupled with relatively low atmospheric CO₂ concentrations, may have played an important role in driving Himalayan glaciation. Further evaluation of Himalayan glacier chronology could yield greater insights into the dominant drivers of Earth’s ice-age climate cycles.
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CHAPTER 1

THE PROBLEM

One of the great unsolved questions in paleoclimatology is the cause of ice ages. We lack a complete understanding of why the earth switches between glacial and interglacial modes of climate. Therefore, our fundamental understanding of the climate system is incomplete. The climate system alternates between periods of global glaciations and interglacial warm periods, with the most recent glacial period culminating with the last glacial maximum (LGM) between ~26,000 and ~19,000 ka (Clark and Mix, 2002). Despite decades of work, a complete solution for ice ages has yet to be proposed.

Mountain glaciers provide essential insight into the dynamics of the Earth’s climate system due to their high sensitivity to temperature and precipitation changes (Schaefer et al. 2008). Evidence for glacial fluctuations is often preserved on the landscape in the form of erosional or depositional landforms. Moraines indicate times at which a glacier has achieved an equilibrium with atmospheric conditions. Constraining the age of these landforms with absolute dating allows us to reconstruct past glacial extent. Developing a global array of glacial records at various latitudes, within different climate settings, will help to isolate significant mechanisms, providing insight to global paleoclimate changes.

Here I present a $^{10}$Be surface-exposure chronology from the Khumbu glacier in the Mount Everest region of Nepal (28°N, 86.8°E). The goal of my thesis is to develop a chronology of moraines constructed during the last glacial period. This glacial system is sensitive to atmospheric heat fluxes originating in the Indian Ocean, and therefore is well-situated to test various hypotheses the role of the tropical system in glacial cycles.
At least two mechanisms have been proposed to exert a dominant control on glacial extent in this area during the course of recent ice-age cycles: 1) local insolation intensity, and 2) the intensity of the summer monsoon. Below, I examine each of these hypotheses and what they would predict for glacial chronology in the Khumbu region.

1.1. Northern Hemisphere summer insolation hypothesis

Variations in Earth’s climate have long been attributed to cyclical changes Earth’s orbital characteristics (e.g., Croll, 1867; Milankovitch, 1941). Milankovitch (1941) proposed that local variations in incoming solar radiation (insolation) drove ice extent. Hays et al. (1976) demonstrated a role for an orbital pacemaker of the climate system on the basis of an absolutely dated reconstruction of past ice volume changes, based on marine benthic δ18O data. A modified version of the Milankovitch orbital theory suggests that high-latitude Northern Hemisphere summer insolation drives the growth of large continental ice sheets over the northern continents, which in turn produce albedo feedbacks that cool the globe (Kawamura et al. 2007; Tierney et al. 2020). The idea of periodic changes in Earth’s orbital geometry had largely been accepted to be in some part controlling Quaternary glacial cycles.

According to Milankovitch theory, more extensive glaciation would be predicted during periods of relatively low local summer insolation intensity, and reduced glacier extent occurs during relatively high insolation intensity. Fig. 1.1 shows local insolation intensity at 28°N for the past 100 ka and the δO¹⁸ record of speleothems documenting fluctuations in monsoon strength. By this insolation signature, classic Milankovitch Theory would predict moraine construction at 74 ka ago and between 22 and 24 ka ago, and reduced glacier extent between 65 and 68 ka ago when insolation was relatively high.
Figure 1.1. Local insolation intensity for 28N. Data is based on calculations presented in Laskar et al. (2004). Yellow bars indicate years of predicted moraine construction.

1.2. Monsoon hypothesis for Himalayan glaciation

A second prominent hypothesis posits that Himalayan glacier extent over Pleistocene ice-age cycles is controlled by the effects of the summer monsoon. There are two versions of this hypothesis. Owens et al. (2002) suggested that past intervals of intensified monsoon activity would result in increased snow precipitation on glacier accumulation areas, driving glacier advance. Conversely, during periods of weakened monsoon intensity, reduced precipitation would lead to glacial recession.

An alternative version of the monsoon hypothesis was introduced by Rupper et al. (2009), who suggested that increased cloud cover during periods of intensified monsoon activity would decrease the amount of incoming solar radiation reaching glacier surfaces, thereby reducing surface melt and stimulating glacier advance. On the other hand, periods of weakened
monsoon activity would yield less cloud cover and therefore allow more solar radiation to reach glacier surfaces, increase ablation, and promote glacier recession.

These hypotheses are not mutually exclusive. Both hypotheses would predict that intensified monsoon activity would favor glacial advance and that a weakened monsoon would lead to reduced ice extent. Because monsoon intensity is directly correlated with NH summertime insolation intensity, the monsoon hypothesis for Himalayan glaciation predicts a chronology opposite to that predicted by the Milankovitch hypothesis. Fig. 1.1 shows local insolation intensity at 28°N for the past 100 ka and the δ¹⁸O record of speleothems documenting fluctuations in monsoon strength. Specifically, if the monsoon hypothesis is correct, periods of moraine construction would be predicted during periods of relatively intense monsoon activity, reported higher δ¹⁸O values, ~18 ka, ~22 ka, ~34 ka, ~38 ka, ~60 ka, and ~68 ka, and periods of ice reduction would be predicted during weak phases of the monsoon at ~21 ka and ~58 ka ago.
Figure 1.2. Chinese speleothem records sourced from the Hulu and Dongge caves. Data from Cheng et al. 2016 are plotted in purple. The $\delta^{18}$O record represents the relative strength of the Asian monsoon, with higher peaks indicating times of relative strength in the monsoon. Yellow bars indicate years of predicted moraine construction.

Figure 1.3. Satellite image of the Himalaya. The arrows indicate the trajectory of two major weather system, the Asian monsoon and the mid-latitude westerlies. The field area for this study is marked by a yellow circle.
1.3. Research Objectives

The purpose of this study is to test the above hypotheses by developing a $^{10}$Be surface-exposure chronology of moraines deposited during the Last Glaciation by the Khumbu Glacier in the high Nepalese Himalaya. Specific objectives are as follows:

1. Develop a glacial geomorphological map of landforms constructed by the Khumbu Glacier during the Last Glaciation.
2. Sample glacial boulders associated with Khumbu Glacier moraines for $^{10}$Be surface-exposure dating.
3. Construct a $^{10}$Be surface exposure chronology of the mapped Khumbu Glacier landforms.
CHAPTER 2

BACKGROUND

2.1. Physical Setting

The Himalaya and the Tibetan Plateau are among the most extensively glaciated landscapes outside of the polar regions. Runoff derived from glacial melt in these regions constitutes an important fraction of runoff that supports large downstream populations. Khumbu glacier is located in the high Khumbu Himal at 27.962 °N, 86.826 °E. The glacier catchment includes some of the highest peaks on Earth, including Mt. Everest (8880 m a.s.l.; also known as Chomolungma in Tibetan and Sagarmatha in Nepalese), Lhotse (8,516 m a.s.l.), and Nuptse (7,861 m a.s.l.).

The Himalayan mountain range is influenced seasonally by two primary climate patterns: The South Asian Monsoon in the summer and the mid-latitude westerlies in the winter. The southern and eastern Himalayas experience a pronounced summer precipitation maximum (as much as 600 mm/yr) from moisture advected north from the Indian Ocean by the southwest monsoon. The interaction of the mid-latitude westerlies with the orography results in a winter precipitation maximum (as much as 25 mm/yr) in the far west of the Himalaya. Winter precipitation is derived from the Mediterranean, Black, and Caspian Seas (Benn and Owen, 1998). Snow and ice mass balance gradients along the Himalayas are tied to these climatic gradients (Owen and Benn, 2005).

The Khumbu Glacier is located within the high peaks of the eastern monsoonal Himalaya and is a temperate ‘summer-accumulation’-type glacier, where peak accumulation and peak ablation take place simultaneously on the upper and lower parts of the glacier, respectively (e.g., Ageta and Higuchi, 1984). Ablation on the lower portion of the glacier is dominated by summer
melt driven by turbulent heat fluxes and direct radiative forcing (Steiner et al. 2018; Singh et al. 2019). The glacier extends ~12 km from the upper reaches of the accumulation area at ~7,000 m a.s.l., near the Western Cwm of Mt. Everest, to its terminus at 4,900 m a.s.l. The equilibrium line of the Khumbu glacier is currently 5,700 m a.s.l. near the top of the Khumbu Icefall (Scherler et al. 2011a). The glacier flows at a rate of ~60 cm/yr (Benn and Owen, 1998). The geologic setting for this area is the Mount Everest massif, which was formed by the Indo-Asian collision ca. 50 Mya and subsequent uplift (e.g., Rowley, 1998). Mount Everest and Lhotse are dominantly composed of the ‘Everest Series’ of weakly metamorphosed sedimentary rocks. The lower part of the Khumbu Glacier catchment is dominated by sillimanite-grade gneisses with leucogranite intrusions. The landscape of the Everest massif is heavily modified by glacial erosion, with large-scale glacial erosional features such as deeply incised glacial valleys, arêtes, horns, and cirques.
Figure 2.1. Google Earth imagery of the Khumbu valley. The yellow box outlines the field limits for this study.
2.2. Previous work

Prior work undertaken in the Khumbu Himal has helped establish the glacial history of landforms deposited by the Khumbu glacier. Fushimi (1978) and Iwata (1976, 1984a) recognized three ice-marginal positions: the Lobuche stage, the Thukla Stage and the Pheriche Stage. An additional Thyangboche Stage, occurring in MIS 3 or 4, was suggested on the basis of radiocarbon dates presented by van Williams (1983) and Rothlisberger et al. (1986). Optically Stimulated Luminescence (OSL) dating supports an advance during the Thyangboche Stage for the maximum position of the Khumbu glacier, beyond the previously interpreted maximum of the Pheriche stage (Richards et al. 2001). Richards et al. (2001) proposed the following numerical ages for the local glacial stage: Lobuche stage (1-2 ka), Thukla Stage (10 ka) and Pheriche Stage (18-25 ka). Finkel et al. (2003) presented a ¹⁰Be exposure-age chronology of glacial landforms in the Everest region indicating moraine construction as early as ~430 ka ago (Thyangboche I), and later at ~60,000 ka ago (Thyangboche II), ~24 ka ago (Pheriche I), ~18 ka ago (Pheriche II), ~11-12 ka ago (Chukhung), ~3 ka ago (Thukla), 1-2 ka ago (Lobuche) and during the historical period (~500 yr BP to present). More research in this region of the Himalaya is essential to determine the dominant climate mechanisms responsible for the timing and amplitude of these observed glacial stages.
CHAPTER 3

METHODS

3.1. Glacial Geomorphic Mapping

I created a glacial geomorphological map for the Khumbu Glacier region using the ArcGIS version 10.3.1 software. Landforms were mapped using the symbology of Barrell et al. (2011). Glacial geomorphic interpretations were developed using a combination of field observations and aerial imagery analysis. Landforms were examined in detail in the field. I also examined stratigraphic sections exposed along stream cuts. In the field, I made observations on the morphology, composition, cross-cutting relationships, and the degree of erosion/weathering for each landform within the field area and made hand-drawn sketch maps that summarized these field observations.

Unmanned aerial vehicle (hereafter ‘drone’) imagery was acquired using a DJI MAVIC PRO 2 and the “Map Pilot” software by Maps Made Easy for iPad. I collected vertical photographs over the Dingboche field area at a height of ~120 m above the ground surface, and with 60% overlap among individual images. I then processed the collected imagery using Agisoft MetaShape Professional Edition to produce digital elevation models (DEMs) and orthomosaics. GPS coordinates collected from sampled boulders were used as ground-control points (GCPs) to improve the accuracy of the orthomosaic and DEM.

3.2. Sample Collection

I collected samples for »Be surface-exposure dating following the methods reported in Putnam et al. (2019). I targeted moraines that appeared stable and that showed no evidence of post-depositional erosion, mass-wasting, or anthropogenic modification (such as
quarrying). I selected for sampling large, stable boulders embedded in moraine ridge crests. I avoided boulders on steep slopes and within or adjacent to fluvial channels. Rock surfaces that were exfoliated, pitted, disintegrated, fractured or otherwise strongly altered were avoided. I prioritized sampling surfaces near the tops of selected boulders with level or gently dipping surfaces, and that either featured glacial polish or else showed minimal signs of granular erosion (Figure 3.1).

Samples were extracted from boulders using the wedge-and-shim technique. To accomplish this, I drilled 3-5 holes around the selected sample site and inserted a set of two shims into each hole. Wedges were then inserted between shims in each of the holes. A hammer was used to drive the wedges into the holes and consequently dislodge the sample from the boulder. I aimed for a maximum sample thickness of 4 cm, if possible, to minimize errors associated with thickness corrections.

The geographic coordinates and elevation of each sample were recorded using a Trimble Geo7x, with no fewer than 500 points recorded per sample. These coordinates were then differentially corrected against a stationary Trimble NetR9 base station. I used the “Theodolite” iPad software to measure the topographic shielding at each boulder location. Shielding data were later also calculated using an internal (UMaine) python script (Audet and Putnam, personal comm.) together with an 8m DEM in QGIS. I measured the dip and dip direction for each sampled surface using the “GeoID” software for the iPad. Each sample was photographed multiple times from different angles. Boulder dimensions were measured with a measuring tape.
Figure 3.1. Examples of boulders sampled for cosmogenic surface-exposure dating. (A) Sample DNG-19-10 embedded within the Pheriche moraine. (B) Sample DNG-19-20 embedded within Dingboche V. (C) Sample DNG-19-30 embedded in Dingboche I. (D) Sample DNG-19-36 embedded in Pheriche ground moraine

3.3. \textsuperscript{10}Be processing and analysis

Beryllium-10 samples were processed at the University of Maine Cosmogenic Isotope Laboratory. Rock surfaces in excess of 4 cm or that had other unwanted portions, such as edges, were marked at the time of collection with a pen and trimmed with a saw upon arrival at the lab. I traced an outline of each sample on scaled graph paper and used digital calipers to determine the thickness of each rock sample selected for analysis. Each sample was then crushed and sieved. The 150-710 micron grain-size fraction was then rinsed with water to remove dust and then some samples were boiled in 10\% HCl to remove weathering products. All samples were then subject to froth floatation with lauryl amine and alcohol-based essential oil to separate
quartz from feldspar and other minerals. The remaining quartz-fraction of the sample was subjected to successive leaches in 2-10 % HF/HNO3 solutions. This was done to remove any other minerals in the sample aside from quartz, as well as to etch away outer rinds of the quartz grains that might contain meteoric ¹⁰Be. Depending on the mineralogy of the sample, I also subjected samples to boiling in orthophosphoric acid and magnetic separation techniques to isolate the quartz. The purity of each quartz separate was checked via ICP-OES. If deemed sufficiently clean (ideally <100 ppm each of Al, Ca, Fe, and Ti), sample processing proceeded to clean laboratory steps. Impure samples were treated with repeated HF/HNO3 leaches until quartz yielding clean (or stable) cation and anion concentrations.

I extracted beryllium from clean quartz separates following the established UMaine protocol available at: https://umaine.edu/earthclimate/research/glacial-geology-and-geochronology-research-group/cosmogenicisotope/. Clean quartz samples were weighed, spiked with a medium-level ⁹Be carrier (‘BDH carrier’; 1007 ppm; average blank ¹⁰Be/²⁹Be = ~5e⁻¹⁵), and dissolved in concentrated HF. Once dissolved, the sample solution was evaporated, and the samples were fumed with HClO₄ and HCl to remove fluorides. I then subjected the samples to chromatography steps designed to isolate Be from other anions and cations. Be(OH)₂ fractions were then precipitated and converted to BeO by combusting at ~1000 °C. BeO samples were mixed with niobium and packed into stainless-steel cathode targets. The samples were sent to the Center for Accelerator Mass Spectrometry at Lawrence Livermore National Laboratory for ¹⁰Be measurement.
3.4. Exposure-age determination

For each sample, I calculated $^{10}$Be surface-exposure ages using version 3 of the UW online exposure-age calculator (Balco et al. 2008). Ages were determined using the Putnam et al. (2019) production-rate calibration data set, which is based on radiocarbon-dated glacial landforms in Scotland. I chose this calibration dataset because it affords sea-level high-latitude production-rate estimates for accepted scaling protocols that agree well with a set of globally distributed calibration sites that are all determined from well-dated landforms. Exposure ages are reported using three commonly used scaling methods: “St” (Stone (2000), as modified from Lal (1991)), ‘Lm’ (a time-dependent version of the ‘St’ scaling protocol that incorporates variations in magnetic field intensity), and “LSDn” (the time-dependent scaling method of Lifton et al. (2014) that incorporates magnetic field and solar fluctuations). All ages include corrections for sample thickness and topographic shielding.

$^{10}$Be concentrations in rock can be affected by erosion and/or snow cover. Observations of striae and glacial polish on many samples indicate that negligible amounts of erosion have impacted the sampled surfaces. Furthermore, there is minimal persistent winter snow accumulation at the elevation of the Dingboche/Pheriche moraine belt in the Khumbu Himal. For these reasons, I did not include corrections for erosion or snow cover in age calculations.
CHAPTER 4

GLACIER GEOMORPHOLOGY

The moraine sequence features two primary moraine belts associated with the Khumbu Glacier system: the Dingboche/Pheriche belt, and the Lobuche belt. The Dingboche/Pheriche belt marks the full-glacial Khumbu ice margin, and the Lobuche moraine belt corresponds to Holocene and historical ice-marginal positions near the modern-day glacier terminus. Here, I focus on describing the Dingboche/Pheriche moraine belt.

4.1. Dingboche/Pheriche Moraine Complex

The Dingboche moraine complex is located ~6 km southeast of the modern Khumbu glacier terminus. There are seven moraines comprising the Dingboche/Pheriche moraine complex. There are five moraines outboard the Pheriche Moraine, and are named in closest proximity to the Pheriche Moraine: Dingboche I, II, III, IV, V. The Dingboche moraines have elevations ranging from ~4,300 m to ~4,430 m. Between the outboard moraines there is a relief of 10-15 m between crests. Meltwater channels occur in between moraine segments. All of the landforms have uniform coverage of grass and shrub vegetation (Figure 4.3.). Abundant large boulders are scattered across the moraines and also concentrated in meltwater channels. Boulders are large and generally well-rooted in moraine landforms. Boulder surfaces exhibit pitting, oxidation, and lichen cover.

The innermost moraine, referred to henceforth as the “Pheriche moraine” is the largest in the complex, rising ~100 m from the valley floor on the ice-contact side (west). The Pheriche moraine is associated with several meltwater channels with graded outwash fans that merge with
an inter-moraine meltwater channel (Figure 4.2.). The Pheriche moraine ridge features several large, well-embedded, sub-rounded boulders. Boulder lithologies are dominantly meta-granite. There is consistent lichen cover and oxidation visible on the surface of the boulders.

The Pheriche recessional complex is inboard of the Pheriche moraine and consists of discontinuous recessional moraine ridges and hummocky ground moraine. This complex of recessional landforms is located at ~4,280 m, ~20 m above the Lobuche Stream floodplain on the valley floor. Sections closest to the Pheriche moraine ice-contact slope of the Pheriche moraine have been overprinted by slump deposits. Recessional moraine ridges contain large (2-4 m tall) embedded boulders.
Figure 4.1. Drone imagery of the Dingboche moraine complex. (A) Orthomosaic (9 cm per pixel resolution) and (B) digital elevation model (50-75 cm per pixel resolution).

Figure 4.2. Ground photograph of the Dingboche moraine complex. Vantage facing south-west of the distal side of the moraines.
Figure 4.3. Glacial geomorphic map of the Dingboche moraine complex. Location shown in Figure 2.2.
CHAPTER 5

RESULTS

I processed 32 $^{10}$Be surface-exposure ages out of the 37 samples collected in the field. I obtained two exposure ages from Dingboche I, three from Dingboche II, two from Dingboche III, six from Dingboche IV, five from Dingboche V, 11 from the Pheriche Moraine-Dingboche VI, and three from the Pheriche ground moraine on the valley floor with one age corresponding to Dingboche VII and two to Dingboche VIII. Results of the $^{10}$Be analyses and procedural blanks are given in Table 5.1 and 5.2, respectively. Calculated $^{10}$Be surface-exposure ages are shown in Table 5.3, and plotted on the glacial-geomorphic map in Figure 5.1. Summary statistics for landform ages are given in Table 5.4, and probability density functions, henceforth referred to as “camel plots”, are shown in Figure 5.2.

5.1. Dingboche/Pheriche Moraine Complex

Most moraine ages are internally consistent and form approximately normal distributions. Here, I take the arithmetic mean to represent the moraine age, and I use the standard-error of the mean together with the systematic production rate uncertainty (2.7%) to represent the uncertainty of the landform age. Landform ages are as follows: 15.83 ± 0.12, 17.70 ± 0.57, 29.72 ± 0.63, 36.68 ± 0.47, 38.14 ± 0.43, 67.38 ± 0.56 (statistics are summarized in Table 5.4.). Two ages from the outermost Dingboche I moraine range between 67.0 ka and 67.7 ka and afford an arithmetic mean age of 67.38 ± 0.55 ka. Three ages from the Dingboche II moraine ridge produce an age range of 34.9 ka - 40.5 ka, with a mean landform age of 38.14 ± 0.43 ka. The two ages from Dingboche III are 37.0 ± 0.60 ka and 43.9 ± 0.87 ka, and do not overlap. The older of these two
ages is older than the outboard Dingboche II moraine and is therefore out of stratigraphic order. For this reason, I regard this age as an outlier and exclude it from further analysis. Six ages from Dingboche IV afford an age range of 29.3 ka - 37.3 ka, with the younger date not overlapping with the other ages in the set, and therefore is considered an outlier. With the exception of the single outlier the ages from Dingboche IV yields a mean age of 32.27 ± 3.56 ka. Five ages from Dingboche V range between 28.8 ka and 30.4 ka and yield a mean age of 36.68 ± 0.47. The eleven ages from the Dingboche VI – Pheriche composite moraine yield a range of 17.1 ka - 22.6 ka, and form an approximately normal distribution with the exception of two ages of 20.0 ± 0.28 (DNG-19-32) and 22.6 ka ± 0.4 ka (DNG-19-28) forming secondary peaks that are older than the bulk of the distribution. One sample from the Pheriche ground moraine on the valley floor, Dingboche VII, yields an age of 17.0 ± .32 ka. Two ages from boulders that inboard of Dingboche VII on the Pheriche ground moraine (Dingboche VIII) range from 15.8 ka to 15.9 ka and yield a mean age of 15.83 ± 0.12 ka.
Table 5.1. Results from $^{10}$Be samples analysis. $^{10}$Be calculations assume an erosion rate of 0 cm/kyr and a density of 2.7 g/cm$^2$. The 07KNSTD3110 AMS standard was used for all samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>CAMS Lab No.</th>
<th>Lat.</th>
<th>Long.</th>
<th>Elev. (m asl)</th>
<th>Thickness (cm)</th>
<th>Shielding</th>
<th>$^{10}$Be/$^{9}$Be ± 1s (10$^{-14}$)</th>
<th>[10Be] ± 1 s (10 atoms x g$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DNG-19-01</td>
<td>BE49305</td>
<td>27.887137</td>
<td>86.824491</td>
<td>4333.29</td>
<td>2.24</td>
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<td>27.888178</td>
<td>86.824489</td>
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<td>0.986258</td>
<td>102.260 ± 1.819</td>
<td>139.05 ± 2.47</td>
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<td>0.987302</td>
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<td>143.90 ± 2.58</td>
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<td>86.823891</td>
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<td>0.984194</td>
<td>51.400 ± 0.952</td>
<td>137.94 ± 2.56</td>
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<tr>
<td>DNG-19-08</td>
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<td>27.893068</td>
<td>86.824286</td>
<td>4397.22</td>
<td>1.61</td>
<td>0.987282</td>
<td>79.890 ± 1.512</td>
<td>179.98 ± 3.41</td>
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<tr>
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<td>1.67</td>
<td>0.987293</td>
<td>77.680 ± 1.459</td>
<td>176.01 ± 3.31</td>
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<tr>
<td>DNG-19-10</td>
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<td>27.892312</td>
<td>86.824388</td>
<td>4392.57</td>
<td>0.94</td>
<td>0.987525</td>
<td>78.410 ± 1.421</td>
<td>179.64 ± 3.26</td>
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<tr>
<td>DNG-19-11</td>
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<td>86.824817</td>
<td>4365.98</td>
<td>0.94</td>
<td>0.986149</td>
<td>124.118 ± 1.749</td>
<td>173.27 ± 2.44</td>
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<tr>
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<td>27.890465</td>
<td>86.824880</td>
<td>4371.22</td>
<td>0.84</td>
<td>0.987264</td>
<td>61.570 ± 0.939</td>
<td>140.13 ± 2.14</td>
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<td>62.270 ± 1.150</td>
<td>169.66 ± 3.13</td>
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<td>DNG-19-14</td>
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<td>27.891202</td>
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<td>0.987582</td>
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<td>176.13 ± 2.84</td>
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<td>0.987374</td>
<td>95.690 ± 1.863</td>
<td>212.05 ± 4.13</td>
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<td>DNG-19-17</td>
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<td>0.985393</td>
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<td>183.62 ± 3.92</td>
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<td>DNG-19-18</td>
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<td>27.892690</td>
<td>86.825218</td>
<td>4395.46</td>
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<td>0.987752</td>
<td>139.473 ± 2.576</td>
<td>185.15 ± 3.42</td>
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<tr>
<td>DNG-19-19</td>
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<td>27.893413</td>
<td>86.825229</td>
<td>4398.06</td>
<td>1.18</td>
<td>0.987257</td>
<td>133.790 ± 2.476</td>
<td>186.31 ± 3.45</td>
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Table 5.1. Cont.

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<th>DNG</th>
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<th>94393</th>
<th>86.825344</th>
<th>4424.34</th>
<th>2.04</th>
<th>0.986215</th>
<th>114.900 ± 1.730</th>
<th>324.89 ± 4.89</th>
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</thead>
<tbody>
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<td>DNG</td>
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<td>84424</td>
<td>86.825340</td>
<td>4425.30</td>
<td>1.08</td>
<td>0.986223</td>
<td>144.600 ± 1.692</td>
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<td>DNG</td>
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<td>82562</td>
<td>86.823055</td>
<td>4381.80</td>
<td>1.07</td>
<td>0.960939</td>
<td>63.310 ± 0.746</td>
<td>86.77 ± 1.02</td>
</tr>
<tr>
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<td>81139</td>
<td>86.822783</td>
<td>4379.35</td>
<td>1.37</td>
<td>0.986829</td>
<td>64.330 ± 1.219</td>
<td>87.16 ± 1.65</td>
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<td>80382</td>
<td>86.822677</td>
<td>4380.13</td>
<td>2.27</td>
<td>0.987232</td>
<td>61.114 ± 1.129</td>
<td>85.28 ± 1.58</td>
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<td>DNG</td>
<td>BE</td>
<td>81499</td>
<td>86.822452</td>
<td>4373.85</td>
<td>2.04</td>
<td>0.987360</td>
<td>62.183 ± 1.152</td>
<td>81.86 ± 1.52</td>
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<tr>
<td>DNG</td>
<td>BE</td>
<td>81547</td>
<td>86.822219</td>
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<td>1.69</td>
<td>0.987491</td>
<td>78.350 ± 1.660</td>
<td>107.99 ± 2.29</td>
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<tr>
<td>DNG</td>
<td>BE</td>
<td>82735</td>
<td>86.821968</td>
<td>4362.62</td>
<td>2.03</td>
<td>0.987587</td>
<td>59.210 ± 1.100</td>
<td>81.50 ± 1.51</td>
</tr>
<tr>
<td>DNG</td>
<td>BE</td>
<td>82562</td>
<td>86.821503</td>
<td>4346.20</td>
<td>1.82</td>
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<td>88.13 ± 1.26</td>
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<td>86.821594</td>
<td>4346.90</td>
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<td>0.987458</td>
<td>60.940 ± 1.130</td>
<td>81.99 ± 1.52</td>
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<tr>
<td>DNG</td>
<td>BE</td>
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<td>86.821433</td>
<td>4341.39</td>
<td>1.67</td>
<td>0.976760</td>
<td>68.200 ± 0.942</td>
<td>93.47 ± 1.29</td>
</tr>
<tr>
<td>DNG</td>
<td>BE</td>
<td>82376</td>
<td>86.821061</td>
<td>4331.86</td>
<td>2.47</td>
<td>0.986632</td>
<td>58.970 ± 0.899</td>
<td>80.53 ± 1.23</td>
</tr>
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<td>DNG</td>
<td>BE</td>
<td>83125</td>
<td>86.820645</td>
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<td>1.47</td>
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<td>59.459 ± 1.073</td>
<td>81.19 ± 1.47</td>
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<td>DNG</td>
<td>BE</td>
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<td>86.820250</td>
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<td>1.89</td>
<td>0.983510</td>
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<td>73.68 ± 1.37</td>
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<td>DNG</td>
<td>BE</td>
<td>86460</td>
<td>86.820008</td>
<td>4288.54</td>
<td>1.55</td>
<td>0.982941</td>
<td>49.550 ± 0.920</td>
<td>78.23 ± 1.45</td>
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<td>DNG</td>
<td>BE</td>
<td>87902</td>
<td>86.819991</td>
<td>4286.71</td>
<td>2.18</td>
<td>0.980734</td>
<td>36.770 ± 0.646</td>
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Table 5.2. Data from procedural blanks. The standard, 07KNSTD3110 was used for every sample.

<table>
<thead>
<tr>
<th>CAMS laboratory no.</th>
<th>Sample ID</th>
<th>Carrier Added (g)</th>
<th>Carrier concentration (ppm)</th>
<th>$^{10}\text{Be}/^{9}\text{Be} \pm 1s$ (10$^{-14}$)</th>
<th>$[^{10}\text{Be}] \pm 1s$ (10$^3$) atoms</th>
<th>Average $^9\text{Be}$ current (mA)</th>
<th>Runs</th>
</tr>
</thead>
<tbody>
<tr>
<td>B92</td>
<td>BE47887</td>
<td>0.20521</td>
<td>1007</td>
<td>0.500 ± 0.035</td>
<td>69.0 ± 4.8</td>
<td>19.0</td>
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<tr>
<td>B93A</td>
<td>BE47898</td>
<td>0.20450</td>
<td>1007</td>
<td>0.504 ± 0.047</td>
<td>69.3 ± 6.4</td>
<td>16.6</td>
<td>5</td>
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<tr>
<td>B101</td>
<td>BE48883</td>
<td>0.20600</td>
<td>1007</td>
<td>0.491 ± 0.038</td>
<td>68.1 ± 5.3</td>
<td>19.3</td>
<td>4</td>
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<tr>
<td>B103</td>
<td>BE49153</td>
<td>0.20092</td>
<td>1007</td>
<td>0.517 ± 0.034</td>
<td>69.9 ± 4.6</td>
<td>24.8</td>
<td>3</td>
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<tr>
<td>B107</td>
<td>BE49313</td>
<td>0.20576</td>
<td>1007</td>
<td>0.561 ± 0.042</td>
<td>77.6 ± 5.8</td>
<td>17.6</td>
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</table>
Table 5.3. Calculated exposure ages of samples

<table>
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<th>Sample ID</th>
<th>St age (ka)</th>
<th>Lm age (ka)</th>
<th>LSDn age (ka)</th>
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<tbody>
<tr>
<td><strong>Dingboche VIII-Pheriche ground moraine</strong></td>
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<tr>
<td>DNG-19-35</td>
<td>15921 ± 297</td>
<td>16355 ± 305</td>
<td>16783 ± 313</td>
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<tr>
<td>DNG-19-37</td>
<td>15756 ± 278</td>
<td>16215 ± 286</td>
<td>16612 ± 293</td>
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<tr>
<td><strong>Dingboche VII-Pheriche ground moraine</strong></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>DNG-19-36</td>
<td>16993 ± 317</td>
<td>17332 ± 323</td>
<td>17809 ± 332</td>
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<tr>
<td><strong>Dingboche VI-Pheriche moraine</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DNG-19-22</td>
<td>18427 ± 218</td>
<td>18621 ± 221</td>
<td>19112 ± 226</td>
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<tr>
<td>DNG-19-24</td>
<td>18133 ± 345</td>
<td>18365 ± 350</td>
<td>18852 ± 359</td>
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<tr>
<td>DNG-19-25</td>
<td>17848 ± 331</td>
<td>18105 ± 336</td>
<td>18589 ± 345</td>
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<tr>
<td>DNG-19-27</td>
<td>17134 ± 319</td>
<td>17461 ± 325</td>
<td>17910 ± 333</td>
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<td>DNG-19-28</td>
<td>22615 ± 482</td>
<td>22006 ± 469</td>
<td>22406 ± 477</td>
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<td>DNG-19-29</td>
<td>17070 ± 319</td>
<td>17405 ± 325</td>
<td>17851 ± 333</td>
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<td>DNG-19-30</td>
<td>18578 ± 268</td>
<td>18750 ± 270</td>
<td>19262 ± 278</td>
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<td>DNG-19-31</td>
<td>17481 ± 326</td>
<td>17773 ± 331</td>
<td>18259 ± 340</td>
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<td>DNG-19-32</td>
<td>20011 ± 278</td>
<td>19968 ± 277</td>
<td>20507 ± 285</td>
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<tr>
<td>DNG-19-33</td>
<td>17247 ± 264</td>
<td>17558 ± 269</td>
<td>18037 ± 276</td>
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<tr>
<td>DNG-19-34</td>
<td>17347 ± 315</td>
<td>17648 ± 320</td>
<td>18138 ± 329</td>
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<td><strong>Dingboche V</strong></td>
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</tr>
<tr>
<td>DNG-19-01</td>
<td>29664 ± 596</td>
<td>27934 ± 561</td>
<td>28571 ± 574</td>
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<tr>
<td>DNG-19-02</td>
<td>30300 ± 577</td>
<td>28471 ± 542</td>
<td>29146 ± 555</td>
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<tr>
<td>DNG-19-03</td>
<td>29443 ± 528</td>
<td>27741 ± 497</td>
<td>28371 ± 508</td>
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<td>DNG-19-04</td>
<td>30378 ± 548</td>
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<td>29200 ± 526</td>
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<td>DNG-19-06</td>
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<td>27822 ± 519</td>
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Table 5.3. Continued

**Dingboche IV**

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<tbody>
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<td>DNG-19-08</td>
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<tr>
<td>DNG-19-09</td>
<td>36473 ± 692</td>
<td>34524 ± 654</td>
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<td>DNG-19-10</td>
<td>37080 ± 678</td>
<td>34962 ± 639</td>
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<tr>
<td>DNG-19-11</td>
<td>36328 ± 517</td>
<td>34419 ± 489</td>
<td>35106 ± 499</td>
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<td>DNG-19-12</td>
<td>29301 ± 450</td>
<td>27618 ± 424</td>
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<td>36212 ± 675</td>
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**Dingboche III**

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<tbody>
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<td>DNG-19-14</td>
<td>37000 ± 603</td>
<td>34902 ± 568</td>
<td>35783 ± 583</td>
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<td>DNG-19-16</td>
<td>43916 ± 865</td>
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<td>40866 ± 804</td>
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**Dingboche II**

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<td>DNG-19-18</td>
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<td>DNG-19-19</td>
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**Dingboche I**

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<td>DNG-19-20</td>
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<td>63960 ± 979</td>
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<td>DNG-19-21</td>
<td>67771 ± 805</td>
<td>63962 ± 761</td>
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Table 5.4. Table of selected landform statistics

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<th>Sample ID</th>
<th>Count (samples excluded)</th>
<th>Age range (ka)</th>
<th>Mean age (ka)</th>
<th>Standard error of the mean (ka)</th>
<th>Standard deviation</th>
<th>Peak age</th>
<th>Median age</th>
<th>Interpreted landform age (with external unc.)</th>
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<td>15.8-15.9</td>
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<td>15.8</td>
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<tr>
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<tr>
<td>Dingboche VI-</td>
<td>9 (2)</td>
<td>17.1-18.6</td>
<td>17.6</td>
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<td>0.5</td>
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<td>17.5</td>
<td>17.70 ± 0.57</td>
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<tr>
<td>Dingboche V</td>
<td>5</td>
<td>28.9-30.4</td>
<td>29.7</td>
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<td>29.7</td>
<td>29.72 ± 0.63</td>
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<td>36.7</td>
<td>0.2</td>
<td>0.5</td>
<td>36.5</td>
<td>36.5</td>
<td>36.68 ± 0.47</td>
</tr>
<tr>
<td>Dingboche II</td>
<td>3</td>
<td>37.6-38.4</td>
<td>38.1</td>
<td>0.2</td>
<td>0.4</td>
<td>38.2</td>
<td>38.4</td>
<td>38.14 ± 0.43</td>
</tr>
<tr>
<td>Dingboche I</td>
<td>2</td>
<td>67.0-67.7</td>
<td>67.4</td>
<td>0.4</td>
<td>0.6</td>
<td>67.5</td>
<td>67.4</td>
<td>67.38 ± 0.56</td>
</tr>
</tbody>
</table>
Figure 5.1. $^{10}$Be Chronology of the Dingboche moraine complex.
Figure 5.2. Camel plots of selected landforms. Production rate uncertainty of 2.7% used in error calculations. See Table 5.3 or Figure 5.1. for sample ages.
CHAPTER 6

DISCUSSION

6.1. Glacial Chronology

Glacial-geomorphic mapping and results from $^{10}$Be surface-exposure ages presented in this study indicate that the Khumbu glacier stood at maximum positions within the Dingboche moraine complex at ~67 ka, ~38, ~37 ka, ~29 ka, ~20 ka, and 18 ka. These periods of full-glacial ice-extent correspond to MIS 4, MIS 3, and MIS 2. This chronology suggests the Khumbu Glacier was slightly more extensive during MIS 4 than MIS 2. In addition, exposure dates indicate that ice-surface lowering of ~100m from the top of the Pheriche moraine to the Pheriche recessional landforms occurred between ~18 ka and ~16 ka. We interpret the older peaks in the Pheriche moraine age distribution to indicate multiple periods of moraine deposition at ~22.6 ka, 20 ka, and ~18 ka led to the construction of the composite moraine. Prominent outwash fans and meltwater channels indicate that ablation of the full-glacial Khumbu Glacier was by surface melt and thus sensitive to atmospheric temperature, similar to the primary mode of ablation on the present-day glacier.

6.2. Regional and interhemispheric comparison of glacier chronologies

The $^{10}$Be surface-exposure chronology presented in this study can be compared with previous established chronologies in the Khumbu Himal, where prior workers identified several different glacial stages using relative and numerical dating methods. For reference, the findings of these studies are listed in Table 6.1. The age range for the Pheriche glacial stage is considered
to occur between 18 ka and 25 ka by Richards et al. (2000a), and at 23 ka in Finkel et al. (2003). The timing of the Pheriche glacier stage is further refined by the chronology presented here, which indicates that the Dingboche VI composite moraine was constructed during multiple advances at 22.6 ka, 20 ka, and 17.9 ka. Furthermore, ages of boulders associated with the Dingboche VIII recessional landforms at the base of the Dingboche VI ice-contact slope afford an age of ~16 ka and correspond with the age of the Pheriche II advance identified by Finkle et al. (2003). The chronology presented in this study can additionally confirm moraine formation in the Dingboche moraine complex at 36.7 ka and 38.1 ka that are broadly consistent with the timing of the Thyangboche II stage dated by Finkle et al. (2003) in the nearby Imja Valley.

Table 6.1. Relative chronologies and numerical dates for the glacial successions in the Khumbu Himal

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Thukla 3–4 (late Holocene*)</td>
<td>Thukla I (&lt;5 ka*)</td>
<td>Lobuche (ca. 1–2 ka: OSL)</td>
<td>Historical (&lt;500 yr B.P.)</td>
</tr>
<tr>
<td>Periche (ca. 20 ka*)</td>
<td>Chhukung (ca. 10 ka: OSL)</td>
<td>Lobuche (1 ka)</td>
<td>Thukla (3.6 ± 0.3 ka)</td>
</tr>
<tr>
<td>Thyangboche (ca. 40–50*)</td>
<td>Periche (18–25 ka: OSL)</td>
<td>Chhukung (9.2 ± 0.2 ka)</td>
<td>Periche II (16 ± 2 ka)</td>
</tr>
<tr>
<td>Platform (&gt;150*)</td>
<td></td>
<td>Periche I (23 ± 3 ka)</td>
<td>Periche I (23 ± 3 ka)</td>
</tr>
</tbody>
</table>

Five ages have been previously determined within my study area. The reported ages, study of origin and sample type are reported in Table 6.2. Samples in Table 6.2. are organized by location and corresponding geomorphic units. There is general agreement (i.e., within respective uncertainties) between the $^{10}$Be surface-exposure ages presented in this study and the exposure ages reported from OSL samples from the Dingboche VI-Pheriche moraine presented by Richards et al. (2000a). Additionally, there is agreement between the exposure ages presented in
this study and the ~17 ka $^{10}$Be surface-exposure age presented in Aoki and Imamura (1999) corresponding to the units Dingboche VIII/VII. The 21.9 ± 3.0 ka sample collected from Richards et al. (2000a) may be supported by the findings of this study, however imprecision in reported geographic positions makes direct comparison difficult. The age of 12.9 ± 0.84 ka determined by Aoki and Imamura (1999) is considerably younger than the ages reported in this study and is therefore inconsistent with the chronology presented here.

Table 6.2. Comparison of sample ages produced in previous studies in the Dingboche moraine complex

<table>
<thead>
<tr>
<th>Dingboche VIII/VII</th>
<th>$^{10}$Be sample- Aoki and Imamura (1999)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(15.83 ± 0.12 ka)/ (16.99 ± 0.32 ka)</td>
<td>16.9 ± 0.75 ka</td>
</tr>
<tr>
<td>Dingboche VI- Pheriche Moraine</td>
<td>OSL sample- from Richards et al. (2000a)</td>
</tr>
<tr>
<td>(Composite ridge: 17.92 ± 0.91 ka; ~20 ka; ~22 ka)</td>
<td>18.7 ± 5.6 ka</td>
</tr>
<tr>
<td>Dingboche Complex</td>
<td>OSL sample- from Richards et al. (2000a)</td>
</tr>
<tr>
<td>(29.72 ± 0.63 ka)</td>
<td>21.9 ± 3.0 ka</td>
</tr>
</tbody>
</table>

The $^{10}$Be surface-exposure ages presented in this study can be compared with chronologies being constructed using the same field and laboratory approach in the Southern Hemisphere, permitting an interhemispheric evaluation of the timing of moraine construction during the Last Glaciation. In particular, numerous $^{10}$Be chronologies have been constructed from the Southern Alps of New Zealand, which are positioned to the south of the Indo-Pacific
warm pool, and therefore provide a useful comparison to the Khumbu Glacier chronology. The composite chronology of Denton et al. (2021; and references therein) for glacier advances in the Southern Alps and the chronology presented here are plotted for comparison in Figure 6.1. There is strong agreement between the timing of glacial activity in the Khumbu glacier and the glacier systems in New Zealand, shown in Table 6.3. There is direct overlap between ages of moraines being constructed in Dingboche during $17.9 \pm 0.9$ ka and $36.6 \pm 0.4$ ka with the ages observed in New Zealand of $18.11 \pm 0.34$ ka and $36.6 \pm 0.9$ ka.

![Figure 6.1](image)

**Figure 6.1.** Comparison of Northern and Southern Hemisphere glacial chronologies. These records are plotted against the glacial inferred temperature record.
This study (ages in ka)

Dingboche Moraine ages

<table>
<thead>
<tr>
<th>New Zealand Moraine Ages cited within Denton et al. (2021) (ages in ka)</th>
</tr>
</thead>
<tbody>
<tr>
<td>17.9 ± 0.9</td>
</tr>
<tr>
<td>20.0 ± 0.3</td>
</tr>
<tr>
<td>22.6 ± 0.5</td>
</tr>
<tr>
<td>27.0 ± 0.6</td>
</tr>
<tr>
<td>29.7 ± 0.6</td>
</tr>
<tr>
<td>32.5 ± 0.8</td>
</tr>
<tr>
<td>36.6 ± 0.4</td>
</tr>
<tr>
<td>36.6 ± 0.9</td>
</tr>
<tr>
<td>41.76 ± 0.9</td>
</tr>
<tr>
<td>44.0 ± 1.1</td>
</tr>
<tr>
<td>67.4 ± 0.6</td>
</tr>
<tr>
<td>65.1 ± 1.4</td>
</tr>
</tbody>
</table>

Table 6.3. Comparison of the Khumbu Glacier and Southern Alps moraine chronologies. There is a strong agreement among the ages of moraine construction within the Dingboche moraine complex and with moraine construction within Last Glaciation landforms in the Southern Alps of New Zealand.

6.3. Can insolation forcing explain the Khumbu Glacier record?

The chronology presented here can be compared with the signature of local insolation to test role of insolation forcing in driving the last glacial cycle in the Himalaya. The classic Milankovitch (1941) theory, which posits that local insolation intensity dominates glacier behavior, would predict moraine construction at ~74 ka ago and ~24 ka ago, when 28°N
insolation intensity was low. The Khumbu Glacier chronology indicates that although the composite Pheriche moraine formed when local insolation was low between ~18 and 22 ka, the glacier also constructed several moraines between 38 ka and 29 ka when insolation was relatively high (Figure 6.1.). Therefore, this chronology indicates that the Khumbu Glacier maintained its full-glacial volume throughout an entire insolation cycle. I interpret this mismatch between the Khumbu Glacier chronology and local insolation to indicate a negligible role for insolation in driving Himalayan glaciation.

Furthermore, the Khumbu Glacier chronology can be used to test the modified version of Milankovitch Theory that would suggest that high-latitude boreal summertime insolation drove the growth of the large Northern Hemisphere ice sheets, and that the increased reflectivity produced by the ice sheets cooled the Northern Hemisphere – if not the globe. Again, although the Pheriche moraine was constructed when Northern Hemisphere ice sheets attained maximum ice volume at ~22-20 ka. However, the Khumbu Glacier chronology also features moraines that were constructed in MIS 4 and MIS 2 - well before the large Northern Hemisphere ice sheets swelled to maximum volumes. Altogether, the Khumbu Glacier chronology suggest that neither insolation forcing nor ice-sheet feedbacks are sufficient for explaining the timing of Khumbu Glacier moraine construction.

6.4. Did monsoon activity drive glaciation in the eastern Himalaya?

Here, I compare the Khumbu Glacier chronology to a paleo-monsoon reconstruction to test the hypothesis that the strength of the Asian Monsoon drives local Himalayan glaciation. The monsoon hypothesis posits that increased precipitation and/or decreased cloud cover associated with intensified monsoon activity drives glacial advance in the eastern Himalaya. If
the monsoon hypothesis is correct, periods of moraine construction would be predicted during periods of relatively intense monsoon activity. Therefore, the monsoon hypothesis would predict maximal glacier extents and moraine construction at ~10 ka, ~60 ka, and ~85 ka, and periods of ice reduction at ~21 ka and ~70 ka ago. However, the Khumbu Glacier chronology indicates episodes of moraine formation at ~18 ka, 29 ka, ~36-38 ka and ~67 ka, during periods of notably weak monsoon intensity. Therefore, there does not appear to be a strong relationship between monsoon intensity and the timing of Khumbu Glacier advances.

6.5. What drives glaciation in the monsoonal Himalaya?

The chronology for the Khumbu Glacier during the Last Glaciation raises a series of notable questions. First, if insolation is important for glacial cycles – why then did the Khumbu Glacier maintain its full-glacial extent throughout the course of an insolation cycle? Second, if Northern Hemisphere ice sheets were important for amplifying the effects of insolation, why then did the Khumbu Glacier achieve and maintain a maximum extent many thousands of years before the ice sheets? These questions highlight that there must be another important heating agent responsible for the growth and recession of eastern Himalayan glaciers. In this regard, there is a noteworthy similarity between Indian Ocean sea surface temperatures (SSTs) and the chronology of moraine construction presented here (Figure 6.1). Indian Ocean SSTs were depressed by ~3.5°C during the much of the period of Khumbu Glacier moraine formation in MIS 3 and MIS 2 (Mohtadi et al., 2018). Furthermore, the onset of SST warming during the last glacial termination corresponds to the onset of ice-surface lowering at ~18 ka. In addition, close agreement between Southern Alps and Khumbu moraine chronologies, which straddle the IndoPacific Warm Pool to the north and south, respectively, supports a role for an important role
for the tropical ocean in driving glaciers in these locations. I interpret this similarity between reconstructed Indian Ocean SST records and moraine formation in the Khumbu and New Zealand chronologies to suggest that heat fluxes off of the Indian Ocean may have been important for controlling the extent of glaciation in the Himalaya.

In addition, atmospheric CO$_2$, an important greenhouse gas, maintained generally low levels during much of the period at which the Khumbu Glacier was at its maximum extent (Figure 6.1). I take this to indicate a potentially important role for CO$_2$ in regulating Himalayan glaciation. Taken together, I suggest a tentative hypothesis that tropical heat fluxes, coupled with CO$_2$ concentrations may have been important factors in driving Himalayan glaciation.
Figure 6.2. Comparison of Paleoclimate indicators during the past 100 ka. (A) Exposure ages plotted against sample elevation. (B) Indian Ocean SST from Mohtadi et al. 2014. (C) Composite atmospheric CO$_2$ record from ice core records (D) Hulu Cave isotope record from Cheng et al. 2016. (E) Local June 21 insolation at 28°N from Lasker et al. (2004).
CHAPTER 7

CONCLUSIONS

Thirty-two high-resolution $^{10}$Be surface exposure ages, underpinned by glacial-geomorphic mapping based on drone imagery and field observations, place chronological constraints on moraines constructed by the Khumbu Glacier during the last ice age. According to the $^{10}$Be chronology, moraines corresponding to the full-glacial configuration of the Khumbu Glacier were formed at 67 ka, 38 ka, 37 ka, 29 ka, 22 ka and 18 ka. Glacial boulders embedded in ground moraine and recessional moraines at the valley bottom record ~100 m of ice-surface lowering by 16 ka. The timing of Khumbu Glacier moraine formation overlaps with the global last glacial maximum. The Khumbu termination began shortly after 18 ka, with glacial thinning to the valley bottom well underway by 16 ka. Prevailing hypotheses suggesting important roles for either orbitally modulated insolation intensity, and/or monsoon intensity for Himalayan glaciation, do not adequately explain the chronology presented here. Likewise, the Khumbu Glacier chronology is not easily explained by feedbacks related to the expansion of Northern Hemisphere ice sheets. A remaining outstanding question is: If insolation, ice sheets, and monsoon variability fail to account for the documented pattern of Khumbu Glacier variability, what then is the primary climatic driver of glaciation in the high Himalaya? Surface conditions in the Indo-Pacific warm pool and lower CO$_2$ values may be viable candidates to explain Himalayan cooling and glaciation during the last ice age. Further development of precise and detailed glacier chronologies will help to address these outstanding questions of glacial cycles on the roof of the world.
REFERENCES


Denton, George et al. The Zealandia Switch: Ice age climate shifts viewed from Southern Hemisphere moraines. Quaternary Science Reviews, 2021


APPENDIX A METHODS

This is the standard operating lab procedure for cosmogenic-nuclide sample prep and laboratory processing. The material presented below has been replicated and modified based on a version presented in a thesis presented by Mariah J. Radue in 2018 titled: Glacial History of the Tsagaan Gol- Potanin Glacier Valley, Altai Mountains, Mongolia.

Sampling is most easily carried out in groups of 3-5 people. One person can extract the sample while others make observations in field notebooks. Once the sample is extracted, then the driller can make notes while others set up the GPS and measure boulder dimensions.

1. Identify boulder for sampling. Boulder should be glacially deposited and show no signs of post-depositional movement. Avoid boulders on steep slopes or at the bottom of a steep slope. Also, note any human and fluvial alteration near the boulder.
2. Identify the sampling site on the boulder. Aim to sample from a flat surface on the top of the boulder, dipping less than 30°. If there is glacial polish or striations preserved on the boulder, then try to sample those surfaces.
3. Mark the intended sample location with a permanent marker and take a picture of the unsampled surface with a scale. Then, measure the orientation of the sample surface. Record the dip and dip direction.
4. Don safety protections: eyewear, gloves, and ear plugs/earmuffs.
5. Drill 3-5 holes into the boulder about 10° dipping from horizontal with a carbide 3/8” drill bit, using a concrete-grade drill (e.g., Hilti TE-6A). Make the holes about 5 cm apart and arc around the desired sample location. Apply gentle pressure to the drill to guide the
bit in. If drilling into a low-angle surface, it can help to make a small vertical divot with the drill to start the hole.

6. Clean the holes with a puffer and insert wedges and shims - one wedge and two shims for each hole. Make the flat surface of the shims parallel to horizontal and place the shims so that the tips are flush with the edge of the boulder. Then insert the wedge between the shims, ensuring that the wedge faces are also parallel to horizontal.

7. Using a hammer, drive in the shims steadily. Make sure that the wedges are going in evenly so that all wedges are experiencing the same amount of pressure. You will hear the wedges ring the same pitch if the pressure is even. Continue to hammer until the sample “pops” off the rock. If the sample does not come off, then you may have to remove wedges and start new holes. This frequently happens if the holes are drilled too steeply. Vice grips are useful when removing wedges and shims. If there is enough sample extracted (usually 600-1000 g), continue with the procedure, as outlined below. If not, repeat by drilling new holes adjacent to the extracted sample.

8. Place a Trimble Geo 7x GPS (or antenna) on the sample location and let the GPS record at least 500 points. The GPS can record positions while the remaining steps are completed.

9. Sketch the boulder and surrounding geomorphic features. Make written notes of boulder features, such as polish, striations, exfoliation, lichen, chicken heads, etc, and proximal surface features.

10. Measure the length, width, and height of the boulder (measure height from ground to sample surface on N, E, S, and W sides).
1. Measure the shielding from the sample location using an iPad. Make sure to determine whether any portion of the boulder is shielding the sample. Record in notebook.

1. All information about the boulder (dimensions, rock type, shielding) should be input into the data dictionary in the GPS.

1. On a canvas sampling bag, write the sample name, date, location, and short description of sample on the bag.

1. With the sample bag on the boulder, and a hammer for scale, photograph the boulder from multiple aspects. Then place sample into the bag (only rock fragments with the rock surface preserved).

1. Once the sample and bag are removed, extend a measuring tape to 2 m and place on the ground near the base of the boulder. Then take a video of the boulder with a GoPro mounted on a monopod, making sure to capture every surface of the boulder.

**A.2 Drone Mapping and Map Generation**

*A.2.1 Drone Mapping*

When drone mapping, we use a DJI Mavic 2 Pro quadcopter with a gimble camera. We use the app “Map Pilot” by Maps Made Easy for iPad. Be sure to check local regulations about drone flying before you map.

1. Determine area for drone mapping. While you have access to the internet, create a flight in Map Pilot and save map for offline use.

2. In the field, determine a flying location. The drone can only survey an area with a radius of 2 km from the starting point, so it is most efficient to start flights in the middle of the study area.
3. Set up the drone and controller. Take the case off the camera. Connect the iPad to the console. Put the sun shield on the iPad. Put the parabolic extenders on the console antennas. Turn on the console. Place the drone on the drone carrying case lid.

4. Make sure that there is an SD card in the drone with sufficient memory and a fully charged battery. Turn on the drone and open the DJI app. Wait for the drone to connect with the iPad. Once it has connected, close the DJI app and open Map Pilot.

5. The drone should appear as an arrow on the Map Pilot App. Create a new flight plan that is one battery-life long. This usually includes three transects and 1 km long for each transect (but this will vary depending on chosen altitude). Plan the flights so they are parallel or perpendicular to your entire flight swath. Tap on the iPad to create new point for flight plan, hold the point down to move it, and double tap to delete. Make sure that there is 70% overlap for the pictures and that the drone is flying at 300 m (or the regulated max height for the area in which you are flying).

6. Save the flight plan. Upload the flight to the drone. Turn the console to P mode. Then clear the drone area and press start.

1. The drone flight will take about 12-14 minutes. Make sure that the wind doesn’t change and that it doesn’t rain. If it does start raining, bring the drone home. The drone will fly along the course and a dot will appear on the flight path when the drone takes a picture. Pay attention to if the drone misses a photograph. If there are too many missing images, you will need to re-fly that segment.

1. The drone will return to the home point when the flight plan is complete. When the drone is about 20 m above the home point, take control of the drone by toggling to the S mode on the console. Lower the drone to a person so that they can catch the drone above their head. They
should keep the drone above their head until the drone is shut down (the way to shut down the drone depends on the initial calibrations).

1. Fly as many flights as possible with the available batteries until the entire field area is covered. Make sure to back up flight images on another drive at the end of each day. It is also advisable to periodically swap out memory cards in case the drone crashes and is irretrievable.

A.2.2 Map Generation with MetaShape

The manual for PhotoScan by Agisoft is found at http://www.agisoft.com/pdf/photoscan-pro_1_3_en.pdf and the tutorial followed when creating DEMs and orthomosaics http://www.agisoft.com/pdf/PS_1.3%20Tutorial%20(BL)%20%20Orthophoto,%20DEM%20(GCPs).pdf. Below is a modified version of the PhotoScan tutorial.

1. PhotoScan Preferences: Open PhotoScan “Preferences” dialog using corresponding command from the “Tools” menu. Set the following values for the parameters on the “General” tab:
   - Stereo Mode: Anaglyph (use Hardware if your graphic card supports Quad Buffered Stereo)
   - Stereo Parallax: 1.0
   - Write log to file: specify directory where Agisoft PhotoScan log will be stored (in case of contacting the software support team it could be required)

2. Set the parameters in the GPU tab as following: Check on any GPU devices detected by PhotoScan in the dialog. Check on “Use CPU” option when less than two GPU are used. Set the following values for the parameters on the Advanced tab:
- Project compression level: 6
- Keep depth maps: enabled
- Store absolute image paths: disabled
- Check for updates on program startup: enabled
- Enable VBO support: enabled

3. Add Photos: To add photos select “Add Photos” command from the Workflow menu or click “Add Photos” button located on Workspace toolbar. In the Add Photos dialog browse the source folder and select files to be processed. Click “Open” button. “Load Camera Positions”. At this step, the coordinate system for the future model is set using camera positions. Note: the camera position is included in the picture meta-data, so we do not have to add camera positions. Add all photos from study area into one group.

4. Align Photos: At this stage PhotoScan finds matching points between overlapping images, estimates camera position for each photo and builds sparse point cloud model. Select “Align Photos” command from the Workflow menu. Set the following recommended values for the parameters in the Align Photos dialog:
   - Accuracy: High (lower accuracy setting can be used to get rough camera positions in a shorter time)
   - Pair preselection: Reference + Generic (in case camera positions are unknown – only Generic preselection mode should be used)
   - Constrain features by mask: Disabled (Enabled in case any areas have been masked)
   - Key point limit: 40,000
   - Tie point limit: 4,000
Adaptive camera model fitting: Enabled (to let PhotoScan distortion parameters estimation).

1. Click “OK” button to start photo alignment. In a short period of time (depends on the number of images in the project and their resolution) you will get sparse point cloud model shown in the Model view. Camera positions and orientations are indicated by blue rectangles in the view window.

1. Place Markers: Markers are used to optimize camera positions and orientation data, which allows for better model output. Select the marker on the Reference pane. Then filter images in Photos pane using “Filter by Markers” option in the context menu available by right-clicking on the markers label in the Workspace pane. Now you need to check the marker location on every related photo and refine its position if necessary to provide maximum accuracy. Open each photo where the created marker is visible. Zoom in and drag the marker to the correct location while holding left mouse button. Repeat the described step for every ground control point (GCP). (Note: this step is much easier once an ortho photo of the area is already made. I recommend proceeding without entering the markers, make an orthophoto, import the orthophoto into Google Earth and find the boulder sample sites based on GPS data. Then redo the following steps).

1. Input Marker Coordinates: Finally, import marker coordinates from a file. Click “Import” button on the Reference pane toolbar and select file containing GCP coordinates data in the “Open” dialog. The easiest way is to load simple character-separated file (*.txt) that contain markers name, x-, y- coordinates and height. In “Import CSV” dialog indicate the delimiter according to the structure of the file and select the row to start loading from. Note that # character indicates a commented line that is not counted while numbering the rows. Indicate for
the program what parameter is specified in each column through setting correct column numbers in the “Columns” section of the dialog. Also, it is recommended to specify a valid coordinate system in the corresponding field for the values used for camera center data. Check your settings in the sample data field in “Import CSV” dialog: Click “OK” button. The data will be loaded into the Reference pane.

1. Optimize Camera Alignment: To achieve higher accuracy in calculating camera external and internal parameters and to correct possible distortion (e.g. “bowl effect” and etc.), an optimization procedure should be run. This step is especially recommended if the GCP coordinates are known almost precisely – within several centimeters accuracy (marker-based optimization procedure). Click the “Settings” button in the Reference pane and in the Reference Settings dialog select the corresponding coordinate system from the list according to the GCP coordinates data. Prior to optimization it is also possible to remove the points with the highest reprojection error values using corresponding criterion in “Edit Menu” → Gradual Selection dialog. Set the following values for the parameters in Measurement accuracy section and check that valid coordinate system is selected that corresponds to the system that was used to survey GCPs: Marker accuracy: 0.005 (specify value according to the measurement accuracy).

   a. Scale bar accuracy: 0.001

   Projection accuracy: 0.1

   • Tie point accuracy: 1

1. Click “OK” button. On the Reference pane uncheck all photos and check on the markers to be used in optimization procedure. The rest of the markers that are not taken into account can serve as validation points to evaluate the optimization results. It is recommended to perform the optimization procedure since camera coordinates are usually measured with considerably lower
accuracy than GCPs, also it allows to exclude any possible outliers for camera positions caused by the onboard GPS device failures. Click “Optimize” button on the Reference pane toolbar. Select camera parameters you would like to optimize. Click “OK” button to start optimization process. (For DJI drone cameras it is usually suggested to optimize the rolling shutter).

1. Set Bounding Box: Bounding Box is used to define the reconstruction area. Bounding box is resizable and rotatable with the help of Resize Region and Rotate Region tools from the Toolbar. Important: The colored side of the bounding box indicates the plane that would be treated as ground plane and has to be set under the model and parallel to the XY plane. This is important if mesh is to be built in Height Field mode, which is reasonable for aerial data processing workflow.

1. Build Dense Point: Cloud Based on the estimated camera positions the program calculates depth information for each camera to be combined into a single dense point cloud. Select “Build Dense Cloud” command from the Workflow menu. Set the following recommended values for the parameters in the Build Dense Cloud dialog:

   - Quality: Medium (higher quality takes quite a long time and demands more computational resources, lower quality can be used for fast processing)
   - Depth filtering: Aggressive (if the geometry of the scene to be reconstructed is complex with numerous small details or untextured surfaces, like roofs, it is recommended to set Mild depth filtering mode, for important features not to be sorted out) Points from the dense cloud can be removed with the help of selection tools and Delete/Crop instruments located on the Toolbar.

1. Build Mesh (optional: can be skipped if polygonal model is not required as a final result): After dense point cloud has been reconstructed it is possible to generate polygonal mesh model
based on the dense cloud data. Select “Build Mesh” command from the Workflow menu. Set the following recommended values for the parameters in the Build Mesh dialog:

a. Surface type: Height Field

b. Source data: Dense cloud

c. Polygon count: Medium (maximum number of faces in the resulting model. The values indicated next to High/Medium/Low preset labels are based on the number of points in the dense cloud. Custom values could be used for more detailed surface reconstruction).

d. Interpolation: Enabled

Click “OK” button to start mesh reconstruction.

1. Edit Geometry: Sometimes it is necessary to edit geometry before building texture atlas and exporting the model. Unwanted faces could be removed from the model. Firstly, you need to indicate the faces to be deleted using selection tools from the toolbar. Selected areas are highlighted with red color in the Model View. Then, to remove the selection use “Delete Selection” button on the Toolbar (or Del key) or use “Crop Selection” button on the Toolbar to remove all but selected faces. If the overlap of the original images was not sufficient, it may be required to use “Close Holes” command from the Tools menu at geometry editing stage to produced holeless model. In Close Holes dialog select the size of the largest hole to be closed (in percentage of the total model size). PhotoScan tends to produce 3D models with excessive geometry resolution. That's why it is recommended to decimate mesh before exporting it to adifferent editing tool to avoid performance decrease of the external program. To decimate 3D model select “Decimate Mesh...” command from the Tools menu. In the Decimate Mesh dialog specify the target number of faces that should remain in the final model. For PDF export task or web-viewer upload it is recommended to downsize the number of faces to 100,000 - 200,000. Click “OK” button to start mesh decimation procedure.
1. **Build Texture** (optional; applicable only to polygonal models): This step is not really needed in the orthomosaic export workflow, but it might be necessary to inspect a textured model before exporting it or it might be helpful for precise marker placement. Select “Build Texture” command from the Workflow menu. Set the following recommended values for the parameters in the Build Texture dialog: Mapping mode: Orthophoto Blending mode: Mosaic Texture size/count: 8192 (width & height of the texture atlas in pixels) Enable color correction: disabled (the feature is useful for processing of data sets with extreme brightness variation, but for general case it could be left unchecked to save the processing time) Click “OK” button to start texture generation.

1. **Build DEM**: Digital elevation model can be generated based on the dense cloud or mesh model. Usually first option is preferred, as it provides more accurate results (low-poly model, being used as a source data, may result in inaccurate DEM) and allows for faster processing, since mesh generation step can be skipped. Select “Build DEM” command from the Workflow menu: Coordinate system should be specified in accordance with the system used for the model referencing. At the export stage it will be possible to project the results to a different geographical coordinate system. After DEM generation process is finished, it is possible to open the reconstructed model in Ortho view by double-clicking on the DEM label in the chunk's contents on the Workspace pane.

1. **Build Orthomosaic**: Select “Build Orthomosaic” command from the Workflow menu: Select desired surface for orthomosaic generation process: mesh or DEM, and blending mode.
Pixel size will be suggested according to the average ground sampling resolution of the original images. According to the surface size and the input pixel size the total size of the orthomosaic (in pixels) will be calculated and shown in the bottom of the dialog box. Generated orthomosaic can be reviewed in Ortho mode similar to the digital elevation model. It can be opened in this view mode by double-clicking on the orthomosaic label in the Workspace pane.

1. **Export Orthomosaic:** Select “Export Orthomosaic” → Export JPEG/TIFF/PNG command from File menu. Set the following recommended values for the parameters in the Export Orthomosaic dialog: Projection: Desired coordinate system Pixel size: desired export resolution (please note that for WGS84 coordinate system units should be specified in degrees. Use Meters button to specify the resolution in meters). Export as TIFF.

1. **Export DEM:** Select “Export DEM” → Export GeoTIFF/BIL/XYZ command from File menu. Set the following recommended values for the parameters in the Export DEM dialog. Export as GeoTIFF with WGS84 projection.

### A.3 Quartz Separation

Detailed below are methods for obtaining clean quartz from a whole rock and Be extraction. The preparation of the rock involves both physical and chemical separation methods. The procedure detailed below is a modified version of the Lamont-Doherty Earth Observatory Laboratory methods (http://www.ldeo.columbia.edu/cosmo/methods).

#### A.3.1 Rock Crushing

Safety information: The crushing, grinding, and sieving of rocks produces high amounts of dust, and inhalation of dust particles should be avoided. Review the procedures for operating the ventilation systems for these pieces of equipment and procedures. ALWAYS WEAR A DUST MASK (NIOSH approved, N95), safety goggles, work gloves, long pants, and closed shoes.
1. Ensure that work area and machinery are thoroughly cleaned.

2. Rock samples may need to be cut using a saw to fit in jaw crusher.

3. The samples are crushed into small pieces using a jaw crusher. Use a piece of wood to guide samples into crusher to ensure that they do not fly away.

4. Samples are then crushed using a disk mill. Place the nozzle of the vent into the whole at the top of the box around the disk mill. This will remove the majority of the dust particles from the source area. Crush rock pieces into sand-sized grains (generally < 0.7mm). It is necessary to put the sample through the disk mill numerous times and progressively move the disks closer together to achieve the desired grain size without producing excess fine-grained sediment.

5. The crushed rock can then be put through a column of sieves to sort the sample by grain size. Use 125-710 μm size sieves.

Cleaning: Saws and rock crushing machines should be thoroughly cleaned after each sample. Rinse saws with water and dry them completely afterward. Use methanol to protect the metal pieces from oxidation. Clean the disk mill using a vacuum, air compressor, and small broom or brush. After cleaning the disk mill, turn it on and let it run for a few seconds without putting a sample in and observe to see if grains are in the pan. Clean sieves with a brush and put in a small ultrasonic bath. Then, dry sieves in an oven and inspect them for cleanliness. If grains are still present in sieves, clean further with a brush or air compressor.

A.3.2 Phosphoric Acid Boiling

Samples are boiled in O-phosphoric acid to dissolve a whole host of minerals in many rock types.

1. Check the beakers thoroughly for cracks and clearly label them.
2. Be careful of cross contamination if you are boiling more than 1 sample.

3. Set up the hotplates in the hood with the metal cages, ensuring that the hotplates are steady on the hood floor.

4. Weigh up to 115 g of non-magnetic sample into 1000 ml beakers. Weight the sample directly into the beaker in the fume hood both to avoid inhaling dust and contaminating the lab with dust.

5. While in the fume hood, add some DI-water to each beaker (to keep the dust down). Then, at the sink rinse them thoroughly with DI-water to wash off the fines.

6. In the fume hood, add up to 400 ml of concentrated (85%) O-phosphoric acid to each beaker and cover the beakers with a watch glass. Set the hotplate to about 325°C and bring the samples to a boil. The boiling can be very vigorous at first, so you must stay in the lab until it has reached a steady rolling boil. Make sure that vigorous boiling isn’t causing the beakers to “walk” off the hotplate. After about 1 hour the boiling will become gentle. Boil for 1-2 hours longer or until the volume reaches 300 ml. After a while, usually a total of about 2-3 hours, the rolling boil subsides, and the surface can become quite flat. This a good time to take off the samples.

**CAUTION:** Sometimes when the sample has boiled too long the acid will become very thick and jelly-like. (It seems to happen more with samples that have a lot of fines and organics, such as lichens from the surface of the rocks—another reason to rinse well.) To reduce the amount of lost in thick gel, pour it off before stirring the sample up and suspending it in this dense liquid. If the samples boil for too long, a dense gel can form which can be difficult to remove without losing a lot of sample.

1. Remove the beakers from the hotplate. You can remove the watch glasses, so they cool faster, but then rinse them with DI-water into a container in the hood. Do not squirt water
into the hot acid! Let the samples cool for about an hour. The acid may form a gel around the sample and on the side of the beaker (this film of supersaturated silica solution), which will dissolve during the sodium hydroxide cleaning.

2. Once the beakers are cool (lukewarm is ok), pour off the acid into the Phosphoric Acid waste container. In the hood, squirt down the sides of the beaker with ~200 ml of DI-water and stir the samples with a clean metallic spatula. Allow the samples to settle and decant the water off into the waste container. Then add another 500 ml of DI-water. You can now take the samples over to the sink without risk of inhaling acid fumes. Rinse them 3 more times with DI-water in the sink.

3. Add 300 ml of DI-water to each beaker. In the fume hood, add 100 ml 50% NaOH (sodium hydroxide) to each beaker. The NaOH will dissolve the silicate coating around the quartz grains left by the phosphoric acid leach. Cover the beakers with the watch glasses and boil for ten minutes. (Use the same watch glass for the same sample as before, otherwise thoroughly rinse off any sample grains so as to avoid cross contamination of your samples.)

4. **DO NOT LEAVE THE SAMPLES! At this step the boiling is usually very vigorous and beakers can “walk” off the hotplate!** Start the hotplate at 300°C. If the boiling is too vigorous, reduce the heat. One hour after the samples boil, take the samples off the hotplate. Allow the samples to cool, about 30 minutes. You can remove the watch glasses immediately, rinsing the lids directly into the beaker. Once, cool, pour off the solution into NaOH waste container. Rinse w/ ~100 ml DI-water and pour off into the waste container and then rinse three times with DI-water and in the sink.
5. Either proceed directly to the HF/HNO3 leaching steps or dry the sample in the oven overnight. If you are drying the samples, transfer them to small beakers, combining the same sample into one beaker. Once the sample is dry let it cool, weigh it, and record the weight in the notebook. Cover the sample with parafilm. If you are going directly to the HF step, combine 2 beakers of the sample into each bottle for the leaching step on the shaker table.

Beaker Cleaning: Scrub the beakers in the sink using a brush if necessary and rinse thoroughly so that no sample grains remain in the beakers. If they are really filthy, you can soak them in a soapy solution. Use DI-water for the final rinse. Dry beakers on the drying rack.

A.3.3 Froth Flotation

This method is used to separate feldspar and mica from quartz. It is based on the froth flotation method developed at the PRIME lab (http://www.physics.purdue.edu/primelab/MSL/froth_floatation.html).

Grain size typically 125-710 μm but you should evaluate your sample and select a size that minimizes poly-mineral grains. We have successfully processed 63-125 μm. Quartz with attached feldspars or mica will float, in which case smaller is better. You can froth as much as 300 g in one bottle, otherwise split it into 2 bottles.

Preparation and Pretreatment -- 1% HF leach

1. Record all information in the froth flotation log.

2. Weigh the sample and record the weight (weigh it directly into a tared 2000 ml leaching bottle. Pour the sample in the hood to reduce dust inhalation and lab contamination).

3. Rinse the sample with DI-water to remove dust.
4. Take a small split (<1g) of the rinsed sample with a spatula and place it in a labeled petri
dish for examination under the microscope (it is easier to look at the minerals after the
sample has been rinsed of dust). Set the sample aside to describe while the sample is
leaching.

5. Add 1% HF solution to the jar filling it approximate 2x the depth of the sample. Place it
on the shaker table for 45-60 minutes. Do one sample at a time so the sample isn’t sitting
in the HF solution for too long. You can start the next sample leaching when you begin
frothing the current sample.

6. Meanwhile describe the sample and record this in the log. Roughly estimate the percent
composition of quartz and feldspar and any other significant minerals. If you don’t know
the mineral, at lead describe color, luster, shape, etc.

Frothing Set Up

1. Fill the 10-liter carboy next to the carbonator with the frothing solution: the final should
contain 0.01 ml/1 glacial acetic acid and 0.01 ml/ lauryl amine (surfactant).

2. A concentrated solution is stored in the cabinet below the hood. Add 10 ml of concentrate
per liter of DI-water and mix well (this does not have to be precise).

3. Rinse off the carbonator tube before placing it into the frothing solution in the carboy.
Make sure it is completely submerged. The solution will be sucked into the carbonator
after it’s been dispensed. Keep at lead a few liters in the carboy so the carbonator does
not suck up air.

4. Hard open the CO2 tank. It is pre-set to ~100 psi (it should not exceed 100 psi).

5. Plug in the carbonator. There is no on/off switch.
Frothing Process

1. After 45-50 minutes, decant the 1% HF solution from the sample into a labeled waste container. DO NOT rinse the sample.

2. Keep the sample in the 2L leaching bottle and add a few drops of mineral oil to the sample and swirl it around. All mineral oil seems to work—pine, eucalyptus, tea tree (Do not use vegetable oils. Although they will work, they are impossible to clean up. Mineral oils are aromatic hydrocarbons and will evaporate as opposed to vegetable oils that are long chain fatty acids.

3. Dispense some frothing solution onto the sample. Carefully swirl around the bottle at the same time. Decant the solution with the floating grains into a plastic collection jar or directly into a filter funnel hooked up to the pump. The first 2-3 additions might not work very well but with each repetition the frothing with get “foamier” and more grains will float. The floating minerals will look clumpy, fluffy, and bubbly and after a few repetitions of froth and decanting, the sinking fraction and floating fraction will look distinct. If the frothing seems to slow down yet you can see there is still feldspar to remove, try added more oil. (An easy granitic sample needs 5-10 repetitions. Usually the quartz looks more grayish than the feldspar. Note that usually granite has much more feldspar than quartz so it is normal that the quartz fraction is smaller than the feldspar fraction. Use your original quartz estimate as a guide and if you are unsure, take a split and check under the microscope before you finish.

4. When you think the separation is complete, take a split from the sinking quartz fraction and check under the microscope to see if any feldspar remains. Difficult samples can be
deceiving, so use this as a guide to check what you naked eye sees. Do not finish the sample without looking at this split or you may quit too early.

Once the separation is complete...

1. Take a tiny split from the floating fraction and record what is in it. Take note of any quartz that floated off with the feldspar fraction. It appears that very fine grain quartz can pour off with the floating fraction and in some cases where the quartz yields are small, it will be important to try and reduce this, or to recover it. Also, poly-mineral grains of quartz and feldspars will float.

2. Rinse the floating fraction in the filter with DI-water.

3. Finish filter the floating fraction, neutralize it with baking soda, and pour down the sink.

Sinking and Floating Fractions Quartz & Recovery:

1. Rinse the sinking fraction with D-I water.

2. Proceed to HF/HNO3 leaching

3. Dry the rinsed floating fraction in the filter in the oven.

4. Once dry, transfer it to a plastic bag and weigh it. Record the weight and calculate the sinking fraction wt. (total wt.-floating wt./floating wt.). If your original quartz estimate was good, it should be very close to the sinking (assuming a clean separate).

RECORD ALL WEIGHTS IN THE LOG

A.3.4 Chemical Preparation Steps

The following steps require the use of strong acids that present skin and inhalation exposure risks, and for HF, systemic toxicity. Review the MSDS sheets and any other documentation
provided. Understand the risks associated with handling the chemicals you are working with, the procedures for reducing any risks, and emergency procedures in the event of an accident.

- Always work in a fume hood with the sash as low as is practical.
- Wear safety/splash goggles and use a full-coverage face shield if there is any risk of splashing.
- Wear appropriate gloves: for work with hot acids, use heavyweight (22 mil) neoprene gloves. For work with HF, you must wear HF-resistant gloves- not all materials are HF resistant (for example, latex). Check your gloves regularly for holes and excessive wear and replace as needed.
- You must wear long pants and closed shoes. Shorts, skirts, and open-toed or fabric shoes are not permitted when working with chemicals.
- Know where the eyewash stations, safety showers, spill kits, and tubes of calcium gluconate gel are located. Small spills contained in a hood can be cleaned up. In the event of a large spill or accident, call your institution’s building manager.
- All HF exposures must be treated as medical emergencies. Flush the exposed area with water until medical help arrives.
- All chemical waste is collected in labeled containers and picked up as hazardous waste. Understand the procedures for collecting, labeling, and disposing of your waste.
- Empty bottles must be thoroughly rinsed out. Fill the bottle with water in the hood to avoid breathing vapors, and then rinse out at least 3 times in the sink. Deface the label, and write very clearly on the bottle, “RINSED.”

Hydrofluoric/Nitric Acid Leach:

Samples are leached in a dilute hydrofluoric/nitric acid solution in order to dissolve
minerals other than quartz and to remove meteoric 10Be. Samples are generally leached twice in 1000 ml of a 5% HF/HNO₃ solution and placed on the shaker table, each time for 2 day, and once in a 2% HF/HNO₃ solution in a heated ultrasonic bath for 24 hours. Some samples require additional (4-10) leaching steps before they are sufficiently clean.

**Shaker Table Leach**

You can put ~150 g of sample in a bottle, though this will vary from sample to sample. Most samples dissolve a lot after the first leaching step, but you might want to adjust the amount of sample for samples that don’t dissolve as much at this step.

- For a 5% HF+5% HNO₃ solution- Add 500 ml MQ-H₂O. Then, working in the fume hood, add 50 ml concentrated (49%) HF and 35 ml concentrated (79%) HNO₃, making sure to use the ACS grade bottles. NOTE: ALWAYS ADD WATER FIRST! NEVER ADD WATER TO ACID!

  - Place bottles on the shaker table overnight. Make sure there are no drips of acid on the sides of the bottles. The samples do not need to be on for a full 24 hours. If you put them on in the afternoon, it is okay to change them the next morning. For a 5% HF+ 2% HNO₃ solution, use 875 ml MQ-H₂O, 100 ml HF, and 25 ml HNO₃.
  - In the hood, pour the acid solution into a properly labeled waste container being careful not to pour out your sample.
  - While working in the fume hood, add -1000 ml of MQ-H₂O to each bottle. Shake them vigorously, and then decant the water into the sink, again being careful not to spill any sample. The acid is dilute enough to now work outside of the hood. Rinse the samples
two more times, filling the bottles about a third of the way and shaking them vigorously each time. The vigorous shaking will work to break up weaker feldspar grains.

- Repeat this shaker table leach step until the sample is clean. Ultrasonic Leach in 2% HF + 2% HNO3:
  - Fill the bottle with 800 ml MQ- H2O
  - In the hood, add 30 ml HF and 21 ml HNO3. 
  - Put the lids on tight when putting into the ultrasonic bath.
  - Fill the bath to the brim with water.
  - Turn on both the sonicator. You will need to check the level of the water from time to time. Even without the heat on, the water will evaporate. Keep it full to the brim.
  - Remove the bottles from the bath and allow them to cool for about 30 minutes.
  - Decant the acid into a waste container.
  - Under the hood, fill the bottles with MQ- H2O to rinse in the waster container.
  - As with the shaker table leach, shake these up vigorously, decant into the sink, and repeat for a total of 3-4 rinses.
  - Transfer sample into a very clean and labeled beaker for storage. Dry in oven. When sample is dry, cover with parafilm.
  - Wash your bottles. Make sure you remove all sample grains from the bottles before adding a new sample! Rinse the bottles thoroughly and scrub with brush. You can turn the bottle upside down and forcefully clean off any grains that may be stuck to the bottom and sides. Once your bottles are cleaned, remove all labels and put them away.

A.3.5. Magnetic Mineral Separation
• An initial rough magnetic separation can be achieved by putting the sample through a chute magnet. The grain size generally is between 0.125-0.7 mm. If the samples have been etch, dry them in the oven in a small beaker.

• The non-magnetic fraction attained using the chute magnet is then put through a frantz isodynamic separator (usually 0.5 Amps and a 5-degree tilt) until few magnetic grains remain. This may take two cycles through the frantz. Collect the magnetic fraction in a plastic bag and return the non-magnetic grains to the beaker.

• Cleaning: Clean the chute magnet thoroughly after each sample. Wipe the frantz and the collection cups with the brush and then with the air hose.

A.4 Extraction of Beryllium from Quartz Method

Version: This version was created by Peter Strand, September 2016. It is an adaptation of the following two procedures, modified for the University of Maine cosmogenic isotope laboratory:

1. John Stone’s Be-10/Al-26 method (http://depts.washington.edu/cosmolab/chem) as modified by Brenda Hall.


Where applicable, I’ve included notes from the two methods.

The method that follows is used to separate Beryllium from pure quartz for AMS measurement.

John says: The procedure described below will cope with up to ~10 mg of Fe and 3-5 mg of Ti, assuming the total amount of Al, Be and other metals is less than 3-5 mg. It can be modified to accommodate larger samples by increasing the size of vessels, ion exchange columns, etc.

“ICP” Aluminum check for quartz purity:
26 10 John says: Check the trace-element content of the quartz separate before dissolving it for Al-Be analysis. It is important to obtain low concentrations of Al, Ti, Mg, Ca and alkalis. High Al levels decrease the 26Al/27Al ratio and limit the number of 26Al ions that can be counted. This will reduce the statistical precision of the measurement. High levels of Ti and other trace elements may complicate the chemical separation described below.

Careful quartz clean-up usually (though not always) results in Al and Ti concentrations of <100 ppm. Higher levels of Al may indicate the presence of impurities such as feldspar, muscovite, garnet, or sparingly soluble fluorides from the HF treatment. Note, a 99.5% pure quartz separate containing ~0.5% feldspar still has an Al concentration of ~1000 ppm.

“ICP” Al-Check

- Select and label a set of small, 8ml Teflon ICP beakers, one for each sample.
- Weigh and record the weight of each beaker with lid on.
- With a clean spatula, transfer 0.05-0.35g of sample into the beaker. (0.1 g of sample is a good target)
- Doing this in front of the anti-static machine helps keep quartz grains from being flung about by static.
- Weigh and record the weight of the beaker and sample with lid on.
- Add a small amount of 1% HNO3 with squirt bottle, enough to wet the grains, then cap the beaker. Thoroughly clean the spatula with isopropanol and a KimWipe after each sample.
- Uncap ICP beakers and place on the large hotplate in the fume hood.
- Don the HF safety gear and get a clean 100 ml Teflon reagent beaker. Carefully pour enough
concentrated HF into the reagent beaker for 2-3 ml for each ICP-check beaker.

- Add 2-3 ml of this HF to each sample with a disposable pipette.
- Add 1 ml of 8% H2SO4 to each beaker and set hotplate to ~275 °F. The samples will dry down to a droplet of H2SO4 overnight. Cool the samples.

*John says:* Check the samples for solid material. An opaque, white, crystalline material indicates that the quartz is not clean enough for Be/Al chemistry. Fluffy white bits may indicate garnet. Samples may have a dark material which is probably illmenite or organic material, both of which can be HF-resistant. Illmenite or organic material can be ignored as they will not interfere with the chemistry and will only slightly contribute to the total error via an overestimation of the quartz weight.

- Add 5 ml of 1% HNO3 to each beaker with the repeat pipettor, and then cap beakers. The solutions are now ready for ICP analysis, and should be not be weighed and recorded until immediately before being sent for ICP analysis.
- Weigh and record the weight of the beaker and solution with lid on. \[ \text{weight} = \text{weight of beaker w/ solution} - \text{beaker tare weight} \]
- Transfer to cleaned and labeled ICP-check centrifuge tubes once weighed.

To get ppm of sample, take measured ppm of solution and multiply by weight of solution (here, ~5.1 g). Divide by g in sample.

*Solution*

i.e., 3.6 ppm Al in ICP solution x 5.1 g of ICP solution = ~18 micrograms of Al in ICP solution, obtained from dissolving 0.1 g of rock. Thus, ~180 ppm Al in rock.

To calculate mg in sample, take ppm of sample (from above) and multiple by weight of quartz to be dissolved for chemistry. Divide by 1000.
i.e. 180 ppm Al in sample x 8 g quartz weight = 1.44 mg in sample

**Sample Weighing, Spiking & Blank Preparation**

- Determine the amount of quartz and carrier needed for each sample.  
  - For a batch of 6-11 samples of similar size, prepare 1 process blank.  
  - Label Jars with tape or Teflon marker.

Select a Savillex jar large enough so you only fill the beaker 1/2 full. Estimate space for 5mls HF per gram of sample, the sample itself, and some water.

*Rosanne says: samples <10g use 90ml Savillex, samples ≥10g use 180ml+ Savillex.*

Use the same size beaker for the blank as for the samples. Name the blank based on the batch number (check the master book): B25, B26, etc.

- To reduce static, wrap Al foil around the beakers and use anti-static gate.

- If you are not using the entire sample, make sure the sample is well mixed so that the split taken is representative of the entire sample.

**Weighing Sample**

**METHOD 1: Weighing directly into Savillex jar. (For a sample size < 25g and Savillex jar < 180ml.)**

- Place a clean labeled Teflon jar wrapped w/ Al foil on the analytical balance (the Al foil reduces static).
- Tare the balance.
- Add desired amount of sample to the jar with clean spatula. Record the weight to 4 decimal places.
- Remove the jar from the balance and cover grains with MQ-water.

*Optional weighing method:*

**METHOD 2: Weight by difference. (If the Savillex beaker + Sample will be > 200g.)**
o Wrap the Savillex jar w/ Al foil.

o Weigh the entire sample in its storage container. Record this weight as “Sample + Tare wt.”.

o Empty the entire contents of the container into the Savillex jar.

o Weigh the empty container. Record this weight as “Tare wt.”

o You will calculate your sample weight – “Sample + Tare” – “Tare”.

o Cover the sample w/ MQ-water.

Be very careful not to spill any sample in this transfer, since your sample weight is being determined by weighing the amount removed from the sample container.

o Clean your spatula and work area between samples!

Adding Carrier (9Be)

Since the natural concentration of 10Be in rock is too low to be detected by AMS we add a known amount of 9Be to each sample.

Record the initial weight of the working carrier bottle confirm that it is equal the final weight from the previous use. Remove all the Parafilm before weighing the bottles and invert the bottles a few times to homogenize the solution. When you are finished spiking all your samples, record the final weight of the carrier bottle.

Everyone’s work depends on the integrity of the carrier. NEVER RISK CONTAMINATING THE CARRIER!

o We calculate the amount of carrier added to a sample by weighing the carrier bottle before and after each addition to a sample, rather than directly weighing the amount delivered to the sample.

o Tare the balance.
o Invert carrier bottle a few times to homogenize the solution. Be sure drops of condensation around the lid are taken up and mixed in. Weigh the carrier bottle and confirm that it equals the final weight from the previous use. Record this weight in both the log and your notebook.

o Remove the cap and pipette 9Be carrier into your sample. Use the “Carrier only” 100 – 1000μL pipette and MAKE SURE THE PIPET IS SET AT THE CORRECT VOLUME!

o Immediately recap the carrier bottle and reweigh it. Work quickly, but carefully. Do not leave the carrier bottle open longer than necessary. We want to reduce evaporation as much as possible.

o Check the pipette tip to ensure that the entire amount removed from the bottle, which is what we are weighing, is delivered to your sample and no drops were left behind in the tip.

o If a drop remains in the pipette tip, remove the pipette tip and rinse it out with some MQ-H2O directly into the sample beaker. Discard the tip and use a new tip.

o Reweigh the carrier bottle and record the weight. Calculate the amount of carrier added to your sample as you go along to ensure you have added the amount of carrier you think you have added.

o When finished, check that the carrier bottle cap is screwed on firmly and seal with Parafilm.

o Record all final weights in the Log Book and in your notebook. Record which carrier you used.

o All of the necessary data (sample and carrier weights) must end up in the database and a printed copy should be taped into the lab book.

o Print the chemistry tracking sheet and tape it to the bench in the Al-Be lab.

**Blanks**

*Roseanne says:*
The primary use of blank is to correct the sample 10Be concentration for any 10Be contamination occurring during the sample preparation.

As a general rule, prepare 1 blank per 8-10 samples if all samples are of similar size and are spiked with the same amount of carrier and you expect they will go through the exact same column chemistry. If sample weights should vary by a factor of 3, make up 2 blanks, one to represent small samples and one for large samples, or if you know or even just suspect some of your samples will require more column chemistry, prepare an extra blank.

The blanks are treated exactly like a sample. Use the same size Savillex jar as you used for your samples, rinse the sides down w/MQ-water as you did for the quartz, and add the carrier in the exact same manner. Prepare blanks at the same time you weigh out and spike samples.

Sample Dissolution

SAFETY INFORMATION: You will be using very large volumes of concentrated HF in this step. Follow all safety precautions. Do not work alone in the lab while pouring large volumes of HF. The sample may react upon addition of concentrated HF, so add the HF slowly and use extra caution with a large sample. Do not swirl your samples for a few hours.

Don gloves, sleeve guards, face shield, and apron. Weighed and spiked samples are taken to the hood. In the fume hood, for each sample:

- Add ~5 ml HF per gram of quartz from the bottle-top dispenser. (Reagent A.C.S. grade is ok)
- Screw the caps on the beakers, loosely at first, to allow for any release of gas if the samples are reactive. After a few hours, tighten the lids.

Roseanne says: If quartz is clean, samples will not react when HF is added.

- If you have the time, just let the samples sit until they’re dissolved, rather than putting them on a hotplate. It’s the easiest and cleanest way to handle them. You eliminate having to deal with
condensation on the lids, and the deposition of silica and fluoride salts on the lid. A 5 gram sample will dissolve in about a day while a 50 gram sample will need several days. Swirling them several times/day helps. Make sure the caps are on tightly! Wear full protective gear including face shield when handling the bottles.

You can speed the dissolution up with heat, but first allow the samples to sit overnight before placing them on the hotplate. You can heat them initially with the lids off and at a very low temperature (~125 °F) for a few hours so you are sure they won’t react violently. Then, put the lids on tightly and turn the heat up to ~300 °F. It is the combination of heat and pressure that really speeds things up. It is important that you used a large enough jar so there is enough headspace to accommodate the buildup of pressure.

*Note:* Savillex Teflon melts at 260 °C (500 °F). Keep the temperature below 220 °C (420 °F).

*(The pancake griddles should not get this hot)*

**DO NOT** put Savillex containers on the ceramic hotplate.

If you are measuring Al, this is where you would take a split for stable Al measurement.

Otherwise, continue with the dry down.

**Evaporation & Dry Down w/ HCl**

Once the samples have dissolved, or are nearly dissolved, you will evaporate off all the HF. Fe, Ti, Al, Be, and other ions are left as chloride salts ready for anion exchange clean up.

Drying down the solutions eliminates F- and Si via the reactions:

\[ \text{H}_2\text{SiF}_6(l) \rightarrow (\text{heating}) \rightarrow \text{SiF}_4(g) + 2\text{HF}(g) \]

\[ \text{HF}(l) \rightarrow (\text{heating}) \rightarrow \text{HF}(g) \]

- Open beakers, rinse droplets off of lids into jars with MQ-water
- Place the vessels on the hotplate and evaporate at ~400 °F

*If leaving overnight, turn hotplate down to ~300 °F*
VERY IMPORTANT! Until the sample is completely dissolved, do not spill a drop! If you lose any solution at this point you are preferentially losing 9Be (the carrier). Once the sample has completely dissolved, Be and Be are in equilibrium, and a spill will not affect the 10/9 ratios.

Small vessels that contain < 100 ml will dry down in a day. Larger volumes may take two days or more. Sometimes there are minerals that won’t dissolve which you’ll centrifuge out later. Place a sign on the front lab door indicating that a HF evaporation is in progress.

Chloride Conversion

Once all HF is evaporated, remove the Savillex jars from the hotplate and cool slightly before adding HCl.

Note: HCl tends to splatter when added to a very hot beaker.

Add ~2-3 ml 6M HCl (amount not critical). Wet all sample and dry down again at ~275 °F. Use the larger amount for samples with a very large residue. Rinse down the sides of the beaker with the HCl addition and/or a little MQ-water. The residue should re-dissolve almost instantaneously.

Samples can be moved to recirculating hood after first HCl conversion

Repeat the HCl addition (using ~2-3 ml 6M HCl) and evaporation step 2 more times (for a total of three HCl additions).

Cool the samples completely. Then add 2 ml 6M HCl to each sample. Close the lid, and allow them to dissolve.
Roseanne says: The final solution may be a deep yellow-green color due to FeCl₃. Some samples may also have thrown a fine, powdery white precipitate that will not re-dissolve. This is probably TiO₂. No Al or Be is co-precipitated with the Ti and it can be removed by centrifuging before the anion exchange.

**Anion Exchange Columns**

Rinse 15-ml centrifuge tubes w/ MQ-water and label them w/ sample ID and “Anion”

Transfer the samples to the labeled centrifuge tubes. You can pour it in, or transfer with a disposable pipette. If the sample is thick, sticky and full of residue it is easier with a pipette.

Add another 1 ml of concentrated 6M HCl to the jar as a rinse and transfer to the centrifuge tube. There should be 3 ml in the tube. Samples are now ready for Anion columns.

The anion exchange columns remove FeIII (and some Ti) in the sample.

Resin = AG-1 X8 200-400# mesh. This procedure uses 3 ml of resin (=filled to the 4cm mark)

The anion columns can be reused many times. Inspect the columns before use. When the anion resin gets too old it will take on a darker color and/or contains bubbles in it. If you need to repack columns, follow the procedure for column packing (see Appendix).

*You can reuse the Savillex jars you dissolved your sample in if they are 90 ml or smaller, and if they are clean. Sometimes the digestion leaves black residue behind. This can be wiped out w/ a KimWipe, but should then be followed by a quick leach w/ some dilute HCl or HNO₃ (~5% is fine) on the hotplate. Use a new clean KimWipe for each jar.

**Prep SAMPLES:**

Centrifuge the samples for 10 minutes at 3500 RPM to remove solids.

**Prep COLUMNS:**
o Place waste containers under columns and drain water from columns. Discard water o Examine resin for bubbles and look on top for dirt from previous samples
o Add 15 ml 1.2M HCl (fill headspace 1 1/2 times). This washes the resin 
o Add 9 ml 6M HCl (fill headspace 1x). This conditions of the resin

COLLECT Beryllium fraction:

Anion Exchange Columns

o Place labeled 30 ml Savillex vials (or 90ml Savillex) under columns.
o Load samples with disposable pipette. Use a new pipette for each sample. Drip the solution down the column wall, reaching as far as possible into the column with the pipette. Do NOT pour the sample into the column. Try not to disrupt the top surface of the resin. Allow them to drain through completely.

(Elute the Beryllium fraction with a total of 9 ml 6M HCL added in 3 aliquots – 3x resin volume, allowing the acid to drain through before the next addition.
o Add 1 ml 6M HCL o Add 4 ml 6M HCL o Add 4 ml 6M HCL

Clean columns:
Strip of Fe & Discard (you don’t need to keep this)
o Replace waste containers
o Add 9 ml 1.2M HCl
o Add 9 ml MQ-water
o fill columns with MQ-water, cap, and store

John says: In strong HCl, Fe(III) forms a range of anionic Cl
- - 2- 3- complexes FeCl4 , FeCl5 and FeCl6 ,
which bind tightly to the anion exchange resin. These will form a yellow-brown band at the top of the resin column. Al and Be do not form strong Cl- complexes and elute from the column with the HCl. Some Ti in the form of Ti(IV)Cl62- will bind, but most will drain through as cationic or neutral species, ending up with the Al + Be.

Sulfate Conversions

- Add 1 ml of 0.5M H2SO4 to each Be/Al fraction and dry-down at ~275 °F. This will take ~ 4-6 hours. NEVER EXCEED THIS VOLUME OF H2SO4! The dried residue from this step may turn an alarming dark-brown to black color due to organics which bled from the anion resin. Don’t worry - it will disappear over the next couple of steps.

  Note: Do NOT add peroxide with the sulfuric acid in this step as it will form Cl gas!

- Once dried down, cool the beakers and add 2 drops of ~2% H2O2 (hydrogen peroxide) (if using 30% H2O2, just use the smallest drop you can – though note that 30% H2O2 decreases strength rapidly with time - we used 2 drops of 30% for a year-old bottle). Then add 2-3 ml of MQ-water with disposable pipette. The cakes will begin to dissolve, taking on an amber/gold-red color (TiO[H2O2])2+) if Ti is present. Reheat the vials. The black charry material will disperse and disappear after a while. Dry the samples down again. Red may creep up walls.

- Cool, repeat the H2O2/water (2 drops ~2% H2O2+ 2-3 ml of MQ-water) addition, and dry the samples a second time. At the end of this procedure, the samples should end up either as compact white cakes or small, syrupy droplets of involatile H2SO4. Samples may be slightly yellow. If they remain charry or dark-colored, repeat the peroxide/water addition and dry them down a third time.
Take the samples up in **4 ml of MQ water**, containing a couple drops of **30% H2O2** or trace of 2% H2O2. Warm them a little if necessary to get them back in solution. Don’t risk evaporating too much water - keeping the acid strength low for column loading gives a sharper elution and cleaner Ti-Be cut. The samples are now in ~0.2 M H2SO4, ready for loading on the cation exchange columns. They can be stored indefinitely in this form.

*John says: Ca2+ can be problematic during sulfate conversion (before cation columns) because*

John says: The cation column separates Al, Be and Ti. The column procedure using 2 ml of resin can handle 3-5 mg of Ti, if the total amount of Al and other metals is less than 3-5 mg. The method easily scales up and the volume of resin can be doubled or tripled.

Crystalline calcium sulfate (CaSO₄) may form, which is difficult to re-

**COLUMN SETUP**

**Cation Exchange Columns**

If reusing columns, simply setup and drain, if not reusing columns, follow below:

- Place waste collection cups. Using a disposable pipette, add 2 ml of DOWEX-50 X8 200-400# cation exchange resin to each column. Fill the column with a little MQ-water and before it drains, slurry in a thin suspension of resin. This will immediately slow the dripping, and you can
keep the column full with water while you slowly add more resin to the 2 ml mark. Tapping the column can help to get the dripping started. Be very careful not to trap air bubbles.

**STRIPPING & CONDITIONING RESIN:**

- Strip the resin by filling each column headspace with 3 M HCl (This is 9 ml, equal to 4-5 resin-bed volumes.) Allow it to drain completely.

- Condition first with 9 ml 1.2 M HCl. Drain completely.

- Make up a beaker of 0.2 M H2SO4 containing a few drops of 30% peroxide (or trace of 2% H2O2.) This is 4 parts 0.5 M H2SO4 to 6 parts MQ water

(75 ml MQ-water + 50 ml 0.5M H2SO4 + 3 drops H2O2 works well)

*John says:* Can use roughly 50-50 solution. Accurate volumes are not important; the aim is to match roughly the acid strength of the sample solution.

- Condition the columns by filling the headspace (9 ml) with above solution. Allow it to drain through.

- Discard any leftover conditioning acid in the waste container, and replace it with 0.5 M H2SO4 containing a dash of 2% H2O2. (about 0.5 ml peroxide to 50 ml acid). Remove waste containers and discard waste in acid-waste container.

**ELUTE Ti:**

**Cation Exchange Columns**

- Place labeled 60 ml Nalgene (“Ti/Al Fraction”) bottles under columns.

- Load each sample onto its column using a clean disposable pipette. Ti will form a narrow brown band at the top of each resin bed, and then begin to move down the columns. Allow the sample to run into the resin completely.
o Add 1 ml of 0.5 M H2SO4 w/ trace 2% H2O2 to each beaker as a rinse. Swirl the beakers to pick up any droplets of the original solution left over from the first load. Add the rinse solutions to the columns after they have drained. Allow this to run in completely.

You will add 10 ml (5 bed volumes) of 0.5 M H2SO4 w/ trace 2% H2O2 to each column in three additions (4 ml + 4 ml + 2 ml). If Ti is present, you can see the Ti band move down the resin and elute from the columns. For samples containing Ti but very little Al, the Ti will elute slower and it may be necessary to add another 1 – 4 ml to completely remove Ti.

o Add 4 ml 0.5 M H2SO4 w/ trace 2% H2O2.

o Add 4 ml 0.5 M H2SO4 w/ trace 2% H2O2. Allow first 8 ml to drain through completely.

o Add 2 ml 0.5 M H2SO4 w/ trace 2% H2O2. Drain.

o Add additional 0.5 M H2SO4 w/ trace 2% H2O2 in 1 ml increments to completely remove Ti. o Repeat above step until columns are no longer dripping yellow and eluate is clear

Roseanne says: You can safely elute until the eluate is clear. If the drips are immediately yellow, the column is probably overloaded with Al. Take note of this, but continue on. You will probably have to do a second column to clean up the sample. If you suspect an overload, still add 10 ml of 0.5 M H2SO4 w/ trace 2% H2O2 to the columns.

John says: 12 ml of the sulfuric acid eluent can be run through the columns without risk of losing Be. Yellow drips start with the first Ti - this shouldn’t be immediate upon adding the acid. Drip will go clear when Ti is gone. Do not add >14 ml.

Make a note in your notebook how many mls is took to elute the Ti, how dark or light, narrow or broad the Ti band is, and when it started dripping yellow.

o Remove Ti/Al Fraction bottles

ELUTE Be:
Cation Exchange Columns

- Place 30 ml labeled Savillex teflon vials under each column. Reuse from before.
- Add 10 ml (5 bed volumes) of 1.2 M HCl (“10%” HCl). This will have to be added in 2 lots. There is no need to allow the first to drain completely before adding the second. Allow it to drain through completely.
- Elute Blanks with 12 ml 1.2 M HCl (2 additional mls). With no other ions “pushing” the Be through the column, it takes a little more to get the Be out.

ELUTE Al:

- Replace 60 ml Nalgene (“Ti/Al Fraction”) bottles under columns. Elute Al from the columns with 6 ml (~3 bed volumes) of 3M HCl.

Clean Columns:

- Flush columns with MQ-water
- Fill columns with MQ-water, cap, and store.

Tip: make sure there are clean centrifuge tubes for the next step. Tubes should be washed in dilute nitric - sit about a week, rinsed twice with water and dried in oven.

Dry down

Beryllium Recovery

- Add ~5 drops of 7.5 M HNO3 to each Be sample and dry on a hotplate at 275 F (will take ~8 hrs. If drying overnight, you can put temp slightly lower)
- Label cleaned 15-ml screw cap centrifuge tubes for each sample.
- Once the Be fractions have dried, cool and remove them from the hotplate. The Be fraction should have contracted to a tiny, clear droplet of concentrated H2SO4. Occasionally they will form a small white cake. This usually indicates the presence of either Ti or Al.
John says: If the Be sample is troublesome to dissolve, even with heat, additional acid can be added as an aid. A lot of precipitate that won't dissolve implies a problem, likely calcium sulfate. Intractable samples usually can be dissolved with the addition of a lot of extra acid and heat. Such samples almost certainly will need to go back through cation columns again. Check the Ca ppm in the original ICP check. If the original Ca was low, this problem should never materialize.

**Transfer to Centrifuge Tubes**

- Pipette 2 ml of 1% HNO3 (TM-grade) into each vial. If pure, the Be fractions will dissolve freely. If they don’t, you can warm the vials for a few minutes on the edge of the hotplate with the lid on, or just wait a few hours.
- Carefully pour the solution into a labeled centrifuge tube. Don’t worry if a last drop clings to the floor of the Be beaker, but if its large, you can pick it up w/ a disposable pipette.
- Immediately add another 2 ml of 1% HNO3 into the vial as a rinse, and transfer to the c-tube.

Brenda says: Precipitate, ignite, and pack Al and Be samples shortly before the accelerator run in which they will be measured. Superstition among practitioners hold that Al-and Be-oxides slowly rehydrate if left for weeks or months after baking and will produce lower beam currents. Cathodes packed in advance of a run (or cathodes which have to be stored after a cancelled run) should be stored in the desiccator cabinet in the Al-Be lab.

**Beryllium Hydroxide Be(OH)2 Precipitations & Washes**

You will precipitate the samples two times, and do 3 washes with a pH adjusted water. This step cleans up your sample and gets rid of Boron contamination. You will see your samples get more clear and translucent with each step.
- Add ~ 250 μl NH4OH to the centrifuge tube, cap it, and mix well on the vortex mixer. You should see the white Be(OH)2 precipitate swirling around. Using a clean pipette tip for each
sample, remove ~ 1 μl to check the pH. It should be close to 9. If the pH is below 8.5, add more
NH4OH (add ~30 μl at a time until you reach the correct pH).

Roseanne says: *If you overshoot and the pH is 10, leave it. It’s better a little high than low.*

- Centrifuge for 10 minutes at 3500 RPM.
- **LOOK AT YOUR SAMPLES CAREFULLY AT THIS POINT** and compare the sample Be to
  the blank Be. They should all be the same size.

Roseanne says: *If the samples are larger than the blank, it indicates Al and you probably need to
do a second cation column. If the sample is smaller than the blank, you may have lost Be. But,
before you make this assumption, check the pH of the supernatant. If it is just pH 8, try adding
more NH4OH and bring the pH to 9. Centrifuge again. If it is still small, just proceed to the 2nd
precipitation. This always improves the clarity and often the size of the precipitate. If the
precipitate is still too big (indicating that there is probably Al in the Be fraction) go to the
section on preparing a sample for a second column (Appendix).*

- Pour supernatants back into labeled 22-ml Savillex Teflon. Be careful not to pour out any
  precipitate.
- **Do a 2nd precipitation:** Add 100μl of 7.5M HNO3 to all your samples. Swirl on the vortex
  mixer until precipitate has dissolved completely. Bring the volume up to 5-ml with MQ-water.
  Swirl again on vortex mixer. Re-precipitate Be(OH)2 by adding ~100μl TM NH4OH. Mix well
  on the vortex mixer.
- Centrifuge for 10 minutes again at 3500 RPM. Decant supernatant into same rinse bottle.

Roseanne says: *After precipitating the Be(OH)2, do not let the samples sit around. Always
centrifuge and pour off the supernatant immediately. Impurities in the supernatant may
precipitate out of the solution over time defeating the purpose of precipitation and wash steps.*
**pH 8 RINSES:**

_Roseanne says: This step presumably gets rid of any Boron-10 contamination, an isobar of 10Be._

- Bring solution volume up to 5 ml with the pH 8 adjusted water (pH8 water is MQ-water w/ few drops NH4OH). Swirl on the vortex mixer, centrifuge, LOOK!, and decant the supernatant into the rinse bottles. Do three pH 8 rinses in total.

*Transfer Samples to Quartz crucibles*

**Be(OH)2 Combustion**

- Set up clean crucibles in the Quartz sled, and on the sled, write the sample ID next to the crucible. If possible, do not fill all 10 positions on the sled, and make a diagram in your notebook with the position of each sample in the sled. _Samples located over sled legs may take slightly longer to dry- down on hotplate._

- Dissolve the Be(OH)2 in the C-tubes with 25 μl 7.5M HNO3. _Good place to pause overnight or longer._

- Swirl on the vortex mixer.

- Bring up to speed in centrifuge (3500 RPM @ 0 seconds) to ensure all liquid is collected.

- Transfer to the crucible using the 200 μl pipette. _Reach all the way into crucible with pipette tip, try not to get liquid on sides of crucible_

- With another pipette, add another 25 μl 7.5M HNO3 as a rinse. Pick this up w/ the same pipette you used for the sample transfer, and add this to the crucible. Use a new pipette tip for each sample. Cover all crucibles with lids except the one into which sample is being transferred.

*Hotplate Dry-Down*

After all samples have been transferred to the crucibles, place the sled on ceramic hotplate in the fume hood with crucible and sled covers off. Begin with a low temperature. You want the sample...
to dry on the bottom of the crucible and heating it too fast can result in it drying around the sides making it more difficult to get the sample out of the crucible after its been combusted. Suggested times:

- 200 C for 15 minutes
- 250 C for 75 minutes
- 300 C for 150 minutes
- 350 C for ~30+ minutes (until dry)
- 400 C for 15 minutes (once dry)

Samples will likely form small white cakes at the bottom of crucible. Samples may dry-down without forming white cakes, in which case the sample is very difficult to see. Don’t worry, it is not lost! Assuming all transfers went well, the Be(OH)2 will appear after combustion.

**Cobust Samples**

- Fetch a propane torch from the flammable cabinet,
- Set up torch, ring stand, and crucible tongs in the fume hood;
- Light the torch.
- After removing the crucible covers from the vial, grasp the vial with the tongs about halfway up. Wave the crucible through the flame cautiously at first (if not completely dry, sample may sputter and bubble up if heated too fast). Once the sample begins to glow orange, hold it in the flame for 30 to 40 seconds more. Some samples never glow, in which case 2 minutes is be more than sufficient. Remove it from the heat and place it back in the same spot in the quartz sled in the hood to cool.
- Once cool enough to handle, cover with labeled crucible covers, and store in the recirculating bench for loading. Samples should be loaded as soon as possible after combusting.

*Tip: make sure your cathode loading tools are clean for the next step. The cleaning procedures are on the following page.*
**Equipment List**

- Cathodes
- Drill Rods, #55
- Stainless Spatulas (scrapers)
- Quartz rods
- Niobium powder
- Scooper
- Cathode holder/stand
- Hammer
- Dust Mask
- Ionizer

**Loading Cathodes for LLNL**

Be extremely careful when working with beryllium metal (oxide form). Beryllium is a known health risk and all precautions must be followed when working with it. Always work in the glovebox, and wear a dust mask.

**Label Cathodes**

- Make sure you are using cathodes that have been cleaned, and check each cathode to make sure the hole is centered, and is the correct size. We occasionally get cathodes with holes that are too small. Check this with the drill rod.
- Label the cathode with the sample number, full sample name and LLNL BE#.

**Clean Drill Rods**

- If you are starting with new drill rods, you only need to wipe them down with methanol. If you
are reusing your drill rods, first wipe them off with methanol. You can rinse them with some water, but dry them off immediately, because they rust easily. Then, clean the ends off with some fine sandpaper (400 or 600 grit). Finally, rinse them off again and wipe them down with methanol.

• **Stainless Spatula’s (Scrapers)**
  
o These should be cleaned in a 10% nitric solution overnight. They should also get at least 1 hour in the ultrasonic bath. Pour off the cleaning acid into the 2nd spatula cleaning acid bottle, and then rinse the spatulas thoroughly. Wrap them well with KimWipes and dry them in the oven.

• **Quartz Rods**
  
o These are cleaned in a 20% nitric solution. Follow the spatula instructions. They have their own teflon bottle labelled “glass cleaning”.

**SET UP IN THE GLOVEBOX**

o Wipe down the glovebox with MQ-water thoroughly. o Place ionizer in the glovebox

o Set up your tools on a clean KimWipe.

o Set up the cathode holder on a KimWipe.

o Set up the following waste containers:
  
  • Be-waste plastic bag for used crucibles, KimWipes, and gloves.
  • A waste cup with isopropanol for the used drill rods
  • A waste cup with MQ-water for the used spatulas, quartz rods
  • A waste cup with MQ-water for the used crucible covers

o You will need the tiny scoop and niobium powder and a hammer. Wipe off the hammer with some isopropanol first.
o Put on clean gloves and sleeves, and close the sample door from the inside. You’ll be here for a while, so you might want to grab a stool.

LOADING THE CATHODE

o Place the cathode on the holder

o Fetch the next crucible and take the lid off.

o Add 2 level scoops niobium. You can adjust this up or down for larger and smaller samples.

o Using the quartz rod first, gently mix the niobium into the sample. Once the Niobium is mixed in,

static is usually not a problem, but before that, the Beryllium can be rather flaky.

o Grind the Niobium and Beryllium together, as you would grind something up with a mortar and pestle.

o Using the stainless steel scraper, scrape together the mixture into the bottom.

o Repeat the quartz rod grinding and scraping a few times.

o When the sample is fully homogenized, use the scraper to collect it into the bottom. You can also tap the crucible on your work surface to get it to collect.

o Carefully tilt the crucible on the edge of the cathode, at a 45 deg. angle or so, and gently tap the crucible, and the cathode with the scraper. This will cause it to pour down onto the cathode. If it doesn’t slide right into the hole, simply tap the sides of the cathode. It will.

o Using the drill rod, hammer the sample into the hole. Hammer hard for about 20 taps, then remove the
drill rod, take the sample off the holder and tap it a few times on the holder. Then hammer another 20 pretty hard taps. Repeats the hammer followed by tapping a total 3 times, and finish off with about 10 more gentle taps. ~70 total hammer taps!

- You can gently turn the cathode upside down on the clean KimWipe to check that it isn’t going to spill out.

- Store the cathode in a labeled storage vial, and double check that all your labels are correct.

- Wipe down your work area before loading the next cathode.

- Store samples in desiccator until they are shipped to LLNL.

**Preparing Resin and Packing Columns**

**Resin Preparation**

**Notes:**

- Soak the resin in 6M HCl in the designated bottle. After a few hours, decant the HCl into a waste container. Fill the bottle with MQ-water, shake and decant after the resin has settled. This will take a couple of hours. Do this 2-3 more times so that it is no longer strong acid.

**Packing columns with resin**

- Before filling the columns with resin, fill the column with water and make sure it drips. Usually tapping the column up and down a few times breaks the surface tension and it’ll begin to drip. Or, squirt in a few drops of methanol before adding water.

- Then, fill the column with MQ-water and using a disposable pipette immediately add some resin from the batch soaking in MQ-water. The initial resin will settle onto the frit and immediately slow the water dripping through. Keep the water volume full while you add the resin. The resin should settle out gradually and evenly as it is added thus avoiding air bubbles getting trapped in the resin bed. Continue to add resin to the column until the proper volume is
reached. If you do get air bubbles, fill the column with some water and suck up the resin with the pipette to re-suspend it and usually it will resettle without bubbles.

**You’ve overloaded a column! Preparing samples for a 2nd Cation Column**

- You still have to precipitate the sample. This is the only way to remove the sulfuric acid. So, do that, and note the volume of precipitate.
- Dissolve the BeOH precipitate in 2ml of 3M HCl and transfer it back to a Teflon beaker.
- Then follow the normal procedure for Sulfate Conversions.

**Table A.1. Acid strengths. Chemical**

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Hydrofluoric Acid</th>
<th>Hydrochloric Acid</th>
<th>Nitric Acid</th>
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<tr>
<td>Molecular Formula</td>
<td>HF</td>
<td>HCl</td>
<td>HNO3</td>
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<tr>
<td></td>
<td>H2SO4</td>
<td>NH4OH</td>
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<tr>
<td>Approx. Strength of Concd. Reagent</td>
<td>49.0 %</td>
<td>37.2 %</td>
<td>70.4 %</td>
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<td></td>
<td>96.0 %</td>
<td>56.6 %</td>
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<tr>
<td>Molarity of Concd. Reagent</td>
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<td></td>
<td>18.0</td>
<td>14.8</td>
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</tbody>
</table>

**DOUBLE CATION COLUMN – (4ml resin)**

- Use the larger columns.
- Fill w/ 4 ml resin. *Note, that these columns are marked w/ height in cm., and not volume.*

4 ml is just
under the 6 cm. mark.

**STRIPPING & CONDITIONING RESIN:**

- ~ 18 ml 3 M HCl (Fill to the top twice.) Allow it to drain completely. ~ 18 ml 1.2 M HCl. Drain completely.
- ~ 18 ml 0.2 M H2SO4 containing a trace of 2% H2O2. Drain.

Remove waste trays and discard acid into waste containers.

**ELUTE Ti:**

- Place 60 ml rinsed and labeled (“Ti/Al Fraction”) bottles under columns.
- Load each sample onto its column using a clean disposable pipette. YOU STILL LOAD in 4 ml.
  
  Allow to soak into the resin completely.
- Add 1 ml 0.5 M H2SO4 containing a trace of 2% H2O2 to each beaker as a rinse and add to the column. Add 18 ml of 0.5 M H2SO4 w/ trace 2% H2O2 to each column. It may be necessary to add a further 4-5 ml of 0.5 M H2SO4 to completely remove Ti. Note how much you use.

**ELUTE Be:**

- Place 22 ml labeled Teflon vials under each column. Add exactly 20 ml of 1.2 M HCl ("10%" HCl).

**ELUTE Al:**

- Place 15-ml bottles under each column to collect the Al fraction. Add 12 ml of 3M HCl.
BIOGRAPHY OF THE AUTHOR

Laura Mattas was born on December 11th, 1996 in Saratoga, New York. She graduated from Burnt Hills Ballston Lake Highschool in New York in 2015. Laura received a Bachelor of Science in Earth and Climate Sciences in 2019 at the University of Maine in Orono, Maine. She began her graduate studies at the University of Maine at Orono in the Fall of 2019. Laura is a candidate for the Master of Science degree in Earth and Climate Science from the University of Maine in May 2021.