

**Appendix A: Experimental Methods:****• Sampling**

- Samples were taken from an exposed hillside profiles.
- Cut 0.5-1m into hillside for undisturbed samples
- Sampled at 5cm intervals and homogenized
- Magnetic Susceptibility was measured in the lab in Xi'an

**• Chemical Extraction Methods**

- Extraction of  $^{10}\text{Be}$  from loess samples was done with the following procedure:
- Measure 0.8-1g of crushed loess sample and 0.5mg of  $^9\text{Be}$  carrier (High Purity Be Standard 1000ppm) into a 50mL centrifuge tube.
- Add 3mL  $\text{H}_2\text{O}_2$  (30%) and 3mL Milli-Q water over 30 minutes.
- Leach the sample with 6mL 12N HCl overnight while agitating and heating on hot dog rollers
- Centrifuge samples at 4000rpm for 10 minutes, then pour off the liquid supernatant to labeled Teflon<sup>®</sup> beakers and place on a hot plate in a chemical hood to dry overnight.
- Meanwhile add 15mL 6N HCl to each centrifuge tube (sediment fraction) and shake to mix. Again agitate and heat the samples over moderate heat overnight on hot dog rollers.
- Centrifuge samples again at 4000rpm for 10 minutes, and add the liquid supernatant to the Teflon<sup>®</sup> beakers with the first dried fraction. Rinse the sediment with 15mL Milli-Q water, mix, centrifuge, and add liquid to the beakers. Dry the liquid completely to a residue on a hot plate overnight again.
- Add 10mL of a 2:1  $\text{HNO}_3:\text{HClO}_4$  solution to each sample and swirl to dissolve dried residue, dry solutions on hot plate again. Next, add 10mL of Aqua Regia to each dried sample residue to dissolve and dry on a hot plate again, repeat this 2 more times (make the Aqua Regia fresh before each addition). Lastly, add 10mL 12N HCl to each beaker, mix to dissolve residue and dry completely overnight.
- Run samples through the Fe separation columns (a pretreated anion exchange resin (BIO-RAD AG1-X8) column) to remove Fe. Condition with 0.5N and 8N HCl and then elute and collect the Be+Al fraction (~25mL) in Teflon beakers with 12N and 8N HCl. Elute the Fe with 0.5N HCl and clean the resin with 6N and 0.5N HCl.
- Dry the collected Be+Al fraction, dissolve residue with 0.4M oxalic acid, heat overnight
- Run samples through Be separation columns (a pretreated cation exchange resin (BIO-RAD AG50W-X8) column): Dissolve samples in 0.4M oxalic acid and transfer to 15mL centrifuge tubes. Centrifuge samples at 3000rpm for 5 minutes. After conditioning the columns with 6N HCl, Milli-Q water, and 0.4M oxalic acid, load the liquid supernatant onto the columns. Elute and discard the first fraction with 0.4M oxalic acid, water and 0.5N HCl (~200mL total). Collect the Be fraction with 1N HCl (~80mL) in a Teflon beaker and dry overnight.
- Transfer dried sample solutions to new 15mL centrifuge tubes. Precipitate & purify  $\text{Be}(\text{OH})_2$  at least 3 times with HCl to dissolve and  $\text{NH}_4\text{OH}$  to adjust the pH to 8-9 and heat to precipitate the  $\text{Be}(\text{OH})_2$ . Centrifuge after each precipitation to force the  $\text{Be}(\text{OH})_2$  to the bottom of the tube and remove the liquid supernatant.

Transfer cleaned  $\text{Be}(\text{OH})_2$  to quartz tubes rinsing the centrifuge tubes with Milli-Q water to get all precipitated material. Centrifuge samples at 3000 rpm for 5 minutes and discard the liquid supernatant and dry the gel residue to a powder in a vacuum oven at  $55^\circ\text{C}$  for a couple days or until ready to run in the AMS.

- Combust samples: Place quartz tube with powdered sample inside into a flame for 20-30 minutes to combust sample from  $\text{Be}(\text{OH})_2$  to  $\text{BeO}$ .
- Press sample targets: Transfer  $\text{BeO}$  samples to 1mL centrifuge tubes and add Nb in a 3:1 Nb:BeO ratio by weight and mix very well. For each sample, press the Nb:BeO mixture into a Cu cathode target and store each target in a labeled plastic vial.
- NOTE: the S1 samples were processed by a slightly different method
  - The initial leaching is the same
  - Only one set of columns are used (the Fe separation columns). They are prepped with 50mL 1N HCl. Samples are loaded on the columns, then 10 aliquots of 1N HCl are added (discard the first aliquot, for samples collect aliquots 2-7 and for a blank collect aliquots 3-9, discard the rest), & clean the columns with 6N and 1N HCl
  - Fe is complexed out of solution by adjusting the pH to 8-9 with 30%  $\text{NH}_4\text{OH}$  and then to pH14 with 50% NaOH. The Fe is then centrifuged and removed
  - $\text{Be}(\text{OH})_2$  is precipitated and cleaned in the same way. Combustion and pressing targets are the same too
- AMS
  - For an excellent detailed description of the specifics of how AMS works please see Vogel et al. (1995).
  - The Accelerator Mass Spectrometer (AMS) at the NSF-Arizona AMS Laboratory at the University of Arizona in Tucson, AZ, is a 3MV tandem accelerator built by National Electrostatics Corporation (NEC) in Wisconsin, USA. It is used to measure  $^{14}\text{C}$ ,  $^{10}\text{Be}$ , and  $^{129}\text{I}$  with each run able to measure 40 samples (including 8 standards) in a typical run time of about 15 hours.
    - For more information on the NEC AMS at the NSF-Arizona AMS Laboratory and its applications in research please see Jull et al. (2008)

Jull, A., Burr, G.S., Beck, J.W., Hodgins, G.W., Biddulph, D.L., McHargue, L.R., Lange, T.E., 2008. Accelerator mass spectrometry of long-lived light radionuclides. *Radioactivity in the Environment* 11, 241-262.

Vogel, J.S., Turteltaub, K.W., Finkel, R., Nelson, D.E., 1995. ACCELERATOR MASS-SPECTROMETRY - ISOTOPE QUANTIFICATION AT ATTOMOLE SENSITIVITY. *Anal. Chem.* 67, A353-A359.