Preparing Samples for Neutron Irradiation (valid as of 12/2015)

Note: We irradiate our samples at the USGS TRIGA reactor w/ no Cd shielding unless otherwise requested. Samples are rotated radially in the reactor but not translated.

1) Mineral Separation If you are unsure of how to best separate minerals for your analysis ask the lab manager for workable strategies. Whenever possible, the coarsest size fraction that can be purified should be sought. This depends upon the grain size of the host rock. Typically, the size fraction will be between 50-500 µm. Grains obtained using standard mineral separation methods should be ultrasonically cleaned prior to neutron irradiation to ensure that they are free of dust and heavy liquid residues. Generally hand selection of grains a mineral separate under a binocular microscope is sufficient to produce a high purity (>99%) sample. Ask the lab manager if you are unsure if your materials are suitable before packing them. Ask the lab manager for protocols if you need to acid leach volcanic samples to remove adhering glass.

2) Sample Weights Optimal sample mass (or crystal size) depends upon age and K-content (see McDougall and Harrison, 1999). As a rule of thumb, pack twice as much material as you are likely to need to ensure adequate material can be recovered after unpacking for analysis. Samples should be weighed with a ±0.05 precision balance with as they are packed.

The following are typical sample masses for Noblesse step-heating or laser fusion experiments:

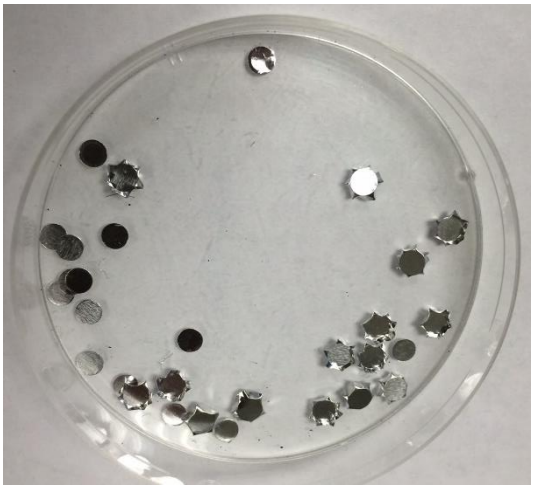
- *K-feldspar* (optimal = 1-2 mg for ~40 steps). Single crystals as small as 400 microns diameter (ca. 250 micrograms) can be successfully step-heated.
- *Muscovite or Biotite* (optimal = 500 micrograms to 1 mg for ~16 steps). Millimeter-size single grains can be step-heated if they are sufficiently old (see below).
- *Sanidine*, *Anorthoclase, Plagioclase* (optimal = 400-600 µm single crystals). Pack about 40-50 crystals per sample. Generally we run about 20 K-rich crystals. For K-poor materials we perform additional analyses as needed to adequately define the isochron plot.

If you are dealing with single crystals and are unsure how small a crystal is feasible to analyze, you may download a spread sheet that will enable you to make a reasonable estimate of signal size as a function of crystal size. ^{40}Ar blanks (typically $5x10^{-16}$ mol) are generally the limiting factor. For IC measurements, > 300 kcps of ^{40}Ar are required to achieve signal/blank ratios > 10. Note that IC signals > 500 kcps incur progressively larger dead time corrections. Signals > 1000 kcps (> 15mV on FC) automatically run in FC-IC mode and are insensitive to blank.

As an example, you can expect a 250 micron diameter, 10 Ma sanidine with 10% K_2O to yield ~$3x10^{-15}$ mol of ^{40}Ar. This signal is feasible for single-crystal IC-only measurements (signal/blank ~ 7) but way too small for FC-IC multicollection. If the same size crystal were 100 Ma, fusion of it would produce $3x10^{-14}$ mol of ^{40}Ar which would be fine for FC-IC multicollection. Similarly, a 10 Ma, 500 micron diameter grain yields nearly $3x10^{-14}$ mol of ^{40}Ar (25 mV of FC signal) that is also fine for FC-IC multicollection (signal/blank > 50).

For incremental heating, a single 100 microgram (1 mm diameter), 10 Ma muscovite would yield an average ^{40}Ar step size of $1.5x10^{-15}$ mol for 10 steps which is three times blank level. Ten steps from a similarly sized 100 Ma crystal would average $1.5x10^{-14}$ mols ^{40}Ar which is 30 times blank level. A 100 micron diameter, 50 micron deep laser ablation pit from the 100 Ma muscovite would be blank level.

3) <u>Packing Foil</u> Ultra-corrosion-resistant, 0.002" thick, alloy 1100 aluminum foil (McMaster 9012K19) is used to pack all samples for irradiation. <u>We no longer use Cu foil due to waste disposal issues</u>. Aluminum is safe and easily unwrapped since it is soft. While we ultimately heat samples in Nb or Ta foil <u>do not irradiate Ta wrapped</u> samples since ^{182}<u>Ta has a 182 day half-life!</u> The base of the Al packet is punched from 8 mm diameter disks to form a 5 mm diameter x 1.5 mm high "pie tin". The top of the packet is formed by a 5 mm diameter disc. The foil (base + lid) is tared before adding the desired mass of sample. The disc is then placed on top of the sample and the sides of the base are folded inward to seal the sample. The sealed sample is then weighed on the tared milligram scale.



4) <u>Sample Identification and Labeling</u> The foil-sealed sample must be appropriately labeled using a ## - ##$ sample identifier **SID** to allow identification when handling. Irradiated samples are identified with the full **SID** on their 0.6 mL storage vials:

SID = 12-40B

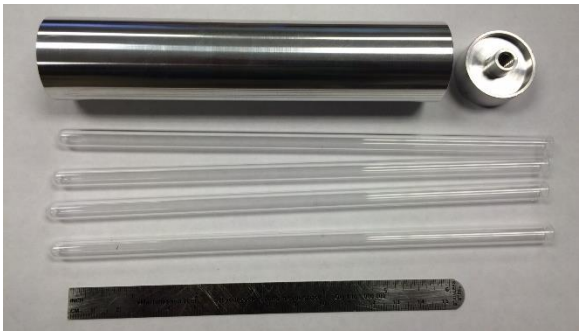
- ## = "**12**" is the number of the irradiation in which the sample was irradiated;
- ##$ = "**40B**" indicates the position (sample 40 from bottom) in tube B.

Due to limited surface area, only the last three characters of the **SID** (40B in this example) are scribed onto the sample itself using a 05 (or finer) permanent marker (Sakura brand works well).

5) <u>Sealing in Quartz Tubes</u> Labeled samples are placed into 6 mm ID - 8 mm OD **quartz** tubes. **Never use borated glass!** Note that a cushion of crumpled Al foil is placed into the tube prior to loading samples. The amount of crumpled foil used depends upon the number of samples being loaded into the tube. Generally 30 to 50 ~1 mm thick samples are loaded per tube. The tube may be filled until the top of the stack is reaches **50 mm** above the base of the tube. The center of the stack of samples should be positioned at **25 mm** above the base of the tube. Generally **6** regularly spaced **flux monitors** are placed in **each** tube. In this example (right) Cu has been used for unknown samples to facilitate contrast with Al wrapped flux monitors. **Use only Al foil**. Other standards are added as necessary. Two additional reference materials (CaF_2 and 22% K2O kalsilite glass) are included with one tube from each irradiation to determine correction factors for nucleogenic Ca- and K-derived Ar. Additional Al foil is added and held in place by a 2 mm diameter **quartz** rod. This rod should have discernable "spring action" that helps prevent samples from sticking together when heated. The tube is sealed with an acetylene torch

under 1 Torr vacuum. The portion of the tube with samples is submerged in water to minimize heating during the sealing process.

6) <u>Irradiation Vessel</u> As many as four sealed tubes are enclosed within a machined aluminum sample holder. Given a maximum number of samples of 50 per tube, this means that up to 200 samples may be irradiated at a time which is more than plenty since we irradiate samples three times a year.



The canister is sealed at the reactor using a sintering process. The base of the canister is 5 mm thick. The centerline of the stack of samples in the tubes is 25 mm. Therefore the centerline of the tubes will be at the centerline of the reactor if the base of the sample canister is positioned 30 mm below the centerline of the reactor.

7) <u>Irradiation Log</u> All samples prepared for irradiation must be entered into an irradiation log file that is delimited by commas (i.e., *.csv file format). There is a single file for each irradiation. <u>A hard copy version of this file is kept in the irradiation log book in Green 056</u>. It is essential that the Tube and Position indicated for the sample correspond to the SID that the sample is labeled with. The *.csv file is read by the data reduction software which creates a directory structure for the entire irradiation and tracks samples based upon the SID. Also note that the program only recognizes certain flux monitor names and mineral/material types. Recognized abbreviations must be used because the program associates these abbreviations with compositions that are used (along with sample masses) to estimate the total mass for each major and selected trace elements for the reactor.

The following information is recorded in eight columns in the *.csv file:

Tube	Position	Name[1]	Mineral[2]	Mass (mg)	Height (cm)	Analyst	Project PI
A	1	FCS	SAN	3.3	1.88	Laboratory	Calibration
A	2	BRNF15-8	MUS	3.0	1.95	O'Brien	Miller
A	3	89BLM123	PHG	4.0	2.00	O'Brien	Miller
A	4	BRNF15-12	MUS	4.2	2.05	O'Brien	Miller
A	5	BRNF15-28	MUS	5.3	2.10	O'Brien	Miller
A	6	89BLM123	PAR	6.9	2.15	O'Brien	Miller
A	7	FCS	SAN	3.0	2.20	Laboratory	Calibration

1. Flux monitor names recognized by software include: Fish Canyon Sanidine (FCS), Bodie Hills Sanidine (BHS), GA1550 Biotite (GAB)

2. Mineral/material abbreviations recognized in software include: Plagioclase (**PLG**), Anorthoclase (ANO), Sanidine (SAN), K-feldspar (KSP), Muscovite (MUS), Phengite (PHG), Paragonite (PAR), Biotite (BIO), Phlogopite (PHL), Amphibole (AMP), Glass (GLS), Whole rock (WRX), Kalsilite Glass (K), Flourite CaF_2 (CA), Halite NaCl (NA), Forsterite (MG).