Georgia Institute of Technology
Terrestrial Cosmogenic Nuclide Geochronology Laboratory

^{10}Be and ^{26}Al Sample Preparation Procedures

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Physical Process

Sample Crushing and Sieving

The objective of this procedure is to prepare the rock sample for the chemical isolation of quartz and extraction of ^{10}Be and ^{26}Al by obtaining the optimum grain size.

Before you begin:

- **Label one large (gallon-size) Ziploc bag with the sample name.**
- **Label three small (quart-size) Ziploc bags with the sample name and the following grain sizes: <250 µm, 250-500 µm, and >500 µm.**
- **Clean the jaw crusher with a wire brush and air.**
- **Clean the disk pulverizer with a wire brush and air.**
- **Clean the sieves with a wire brush. Make sure there are no grains stuck in the sieve openings.**
- **Only open one sample at a time to avoid confusion.**

1. Use the jaw crusher to crush the rock into sand to pebble size pieces that will fit through the chute of the disk pulverizer. Make sure the collection box is under the jaw crusher.

2. On the disk pulverizer, adjust the gap between the grinding plates to about 2 mm. Turn on the dust collector. Close the plate cover and turn on the motor. Make sure the collection box is under the crusher. Feed the sample through the chute.

3. Sieve the sample for the optimum grain size of 250 to 500 µm.

4. If there are any grains above 500 µm in size remaining repeat steps 2 and 3 until the optimum grain size is obtained.

5. Pour the different grain size fractions into the appropriate labeled quart-sized Ziplioc bags. Fold the air out of the small bags and place them into the labeled large bag.

6. Clean the jaw crusher, disk pulverizer, and sieves with wire brushes and air after each sample.

The 250 to 500 µm sample fraction is ready for the HCl leach.

Chemical Process

12-Hour 1:1 (6N) HCl, 0.03% H_2O_2 Leach

The objective of this procedure is to remove (dissolve) carbonates, organic material, and iron oxides from the sample.

Before you begin:

- **Always wear a lab coat, nitrile gloves, and eye protection.**
- **All chemical transfers must be done in the fume hood.**
- **Label a glass beaker of appropriate size (250 mL, 600 mL) with the sample name and weight of the beaker.**
- **Label a large glass beaker (1 L glass beaker if sample size is less than 100 g or 4 L glass beaker if sample size is between 100 g - 300 g) with the sample name and the solution's recipe.**

1. Tare the small glass beaker and add the sample to the beaker. Record the weight of the sample on Worksheet 1 (see page 27).

2. In the HCl fume hood: In the large glass beaker, prepare a 1:1 (6N) HCl, 0.03% H_2O_2 solution. The appropriate 1:1: HCl solution volume is 10 mL of solution per every gram of sample used. The appropriate volume of H_2O_2 is equal to 0.03*sample weight. When preparing the solution, always add NP H_2O first, followed by HCl, and then H_2O_2. Lastly, slowly add the sample in small increments. The solution will strongly effervesce if the sample has a high $CaCO_3$ content. If this happens, wait a few minutes in between adding the sample increments. Be careful not to allow the beaker to overflow.

   An example solution mix for 100 g of sample:
   500 mL of NP H_2O
   500 mL of HCl
   3 mL of H_2O_2

3. In the HCl fume hood: Cover the large glass beaker containing the solution with a watch glass and place it on a hot plate. Heat the solution to a sub-boiling temperature for 12 hours (hot plate temperature setting of ~ 90^{O}C to 125^{O}C depending on beaker size). Set a timer for 12 hours to turn the hot plate off. Make sure the acid does not boil.

4. After heating, allow the acid solution to cool completely before emptying. The sample should be thoroughly rinsed three times with NP H_2O. The first two rinses should be emptied into the waste barrel and the third rinse can be emptied into the sink. Be careful of condensation drips when removing the watch glass. Some of the lighter materials will float off in the rinse stage.

5. Use a 'squirt' bottle to transfer the sample into the small pre-weighed glass beaker and dry the sample in the oven. After drying is complete, weigh the sample and record the sample weight on Worksheet 1.

6. All beakers used for HCl leaching should be cleaned with DI water and detergent (see “Cleaning Procedures” on page 21).

The sample is ready for a series of HF/HNO_3 leaches.

Series of 9-Hour 1% HF / HNO_3 Ultrasonic Leaches

The objective of this procedure is to isolate pure quartz and remove any meteoric ("garden variety") ^{10}Be and ^{26}Al from the sample.

Before you begin:

- **Always wear a lab coat, acid coat, eye protection, face mask, and two pairs of nitrile gloves when handling HF!!**
- **Know what to do in the case of a spill (see Emergency Procedures on page 25).**
- **Label the 4 L HDPE canisters on the top and side with the sample name. Also, label the top of the canister with the solution recipe.**
- **All labeling should be done with a blue Sharpie marker. If acid leaks onto the canister, the blue writing will turn green.**

This procedure is divided into two parts: preparing the leaches and emptying/rinsing the leaches.

Preparing Leaches

1. This process typically involves three HF leaches. The sample will be split into several canisters. For the first leach, no more than 30 g of sample can go into an individual canister (7.5 g of sample per 1 L of solution). For the second and each successive leach, no more than 60 g of sample can go into an individual canister (15 g of sample per 1 L of solution).

2. Record the initial weight of the sample on Worksheet 1. Locate the chart with solution recipes for a 1% HF / 1% HNO_3 solution on the side of the HF fume hood (also see "Solution Recipes" on page 18). Record the appropriate solution recipe on the top of each canister as well as on Worksheet 1 for the 7.5 g sample/L solution.

3. In the HF fume hood: When preparing the solution, always add NP H_2O first, followed by the HNO_3, followed by the HF, and then the sample. Measure the HNO_3 and HF in graduated cylinders inside the fume hood. Pour the HNO_3 from the 500 mL bottle labeled "HNO_3 concentrated" and pour the HF from the 1 L bottle labeled "HF concentrated". Lastly, add the sample in small increments to the canister. As each component is added to the canister, check it off on the solution recipe on the canister lid.

   An example of a 1st HF leach mix:
   3660 mL of NP H_2O
   54 mL of HNO_3
   83 mL of HF
   30 g of sample

   An example of a 2nd or 3rd HF leach mix:
   3660 mL of NP H_2O
   54 mL of HNO_3
   83 mL of HF
   60 g of sample

4. Fill the ultrasonic tanks to ~2 cm from the top with DI water. Make sure that there is not so much water as to overflow the tank. Check that the canister lids are on correctly and place the canisters in the ultrasonic tank. Place labeled canisters filled with H_2O into any unused spaces in the tank to minimize evaporation. Turn on the heater and power for the tanks. The temperature and power should remain at ~ 80ºC to 90ºC. Canisters should remain in the heated/powered tank for 9 hours.

5. After a 9 hour leach, ensure that the lids are on the canisters and carefully move the canisters to the bench top to cool down (CAUTION: canisters will be hot!). Make sure that the power and heat to the tanks are turned off. It is safe to open the canisters when they are at room temperature.

Emptying/Rinsing Leaches

1. After heating, always allow the acid solution to cool to room temperature before opening the canisters. Place a funnel in the liquid waste barrel opening. First, empty the solution into the waste barrel. Then, the canisters should be thoroughly rinsed three times with NP H_2O. The first two rinses should be emptied into the acid waste barrel. The third rinse can be emptied into the sink. Be careful of condensation drips when opening the canister. Work only with one canister at a time and complete one set (one sample) of canisters before moving on to a different sample.

2. Use a 'squirt' bottle to transfer the sample into the pre-weighed small glass beaker and dry the sample in the oven. After drying is complete, weigh the sample and record the sample weight on Worksheet 1.

3. After the 1st leach, allow the sample to dry in the oven and repeat the 1% HF / 1% HNO_3 leach two more times with the 15 g sample/L solution (see recipe on side of HF fume hood or in "Solution Recipes" on page 18).

4. Clean all canisters once with DI H_2O, wipe with a Kimwipe, and rinse again with DI H_2O.

The sample is ready for the addition of Be and Al carrier and quartz dissolution.

Determine Amount of Quartz Needed

The amount of quartz needed for an AMS measurement of ^{10}Be or ^{26}Al concentrations is a function of the sample's estimated age and location. Older samples and ones from high altitudes or high latitudes (higher production rate) will require less quartz than young samples or those samples from low altitudes and latitudes (lower production rate).

You can get a rough estimate of the amount of quartz necessary for an AMS measurement by using the quartz calculators for surface samples, depth profiles, or catchment-wide erosion rates, which can be found at:

http://shadow.eas.gatech.edu/~kfrankel/quartz

Here, you will find Excel spreadsheets and detailed instructions for calculating the amount of quartz needed for each sample. This procedure utilizes the excellent web-based cosmogenic nuclide age calculator and resources that have been made available through the CRONUS-Earth Project at:

http://hess.ess.washington.edu/math/

Once you have determined how much quartz is necessary, proceed to the next step.

Be and Al Carrier

The objective of this procedure is to add a known amount of Be and Al carrier to the sample and dissolve the quartz in solution.

Before you begin:

- **Label a Teflon beaker of appropriate size (250 mL, 500 mL, or 1000 mL) on both sides with the sample name and the weight of the beaker. Use a blue Sharpie marker.**
- **Always wear a lab coat, acid coat, eye protection, face mask, and two pairs of nitrile gloves when handling HF!!**

1. Determine the amount of quartz needed by using the quartz calculator (see above).

2. Tare a Teflon beaker and weigh out the amount of quartz needed on the high-precision balance with doors. Be careful not to spill any sample inside the balance. Record the weight of the quartz on Worksheet 1.

3. If using a weigh boat to measure the sample: Tare the weigh boat and measure the quartz on the high-precision balance with doors. Pour the weighed quartz into the Teflon beaker. Reweigh the weigh boat and subtract out the extra weight (due to the quartz sticking to the weigh boat) from the sample weight. Record the weight of the quartz on Worksheet 1.

4. Tare a 5 mL beaker and weigh 0.3g (0.3mL from the autopipette) of Be carrier on the high-precision balance with doors. Record the weight, concentration, and name of the Be carrier on Worksheet 1. Pour the Be carrier into the Teflon beaker with the quartz. Rinse the 5mL beaker three times with NP H_2O. Note: Assume a density of 1 g/mL for the Be and Al carriers. Make sure to use a new tip for the autopipette for each set of samples.

5. Tare a 5 mL beaker and weigh 2 g (2 mL from the autopipette) of Al carrier on the high-precision balance with doors. Record the weight, concentration, and name of the Al carrier on the worksheet. Pour the Al carrier into the Teflon beaker that contains the quartz and Be carrier. Rinse the 5 mL beaker three times with NP H_2O. Note: The autopipette has a maximum volume of 1 mL. Two grams (2 mL) of the Al carrier is equal to two times the autopipette. Make sure to use a new tip for the autopipette for each set of samples.

6. In the HF fume hood: Add to the Teflon beaker, 0.3 mL of concentrated HNO_3 per every gram of quartz used. Record the amount of HNO_3 added on the worksheet.

   For example:

   If 20 g of quartz is used, then 6 mL of HNO_3 is required.

7. In the HF fume hood: Add to the Teflon beaker, 5 mL of concentrated HF per every gram of quartz used. Add the first 1/3 of the HF to the beaker and make sure that there is no reaction occurring. Then add the remaining 2/3 of the HF to the beaker. Record the amount of HF added on the worksheet.

For example:

If 20 g of quartz is used, then 100 mL of HF is required.

8. Place a Teflon lid on the beaker and place the beaker on a hot plate. The beaker should be heated to a sub-boiling temperature (hot plate setting of ~200 ºC to 350ºC depending on beaker size). Make sure the HF/HNO_3 solution does not boil.

9. After ~2 hours, check to see if all the quartz has dissolved. Be careful of condensation drips when opening the lid. If all the quartz has not dissolved, stir the sample with a Teflon stir rod (be sure to use a different rod for each sample) and replace the lid until all of the quartz has dissolved. After all quartz has dissolved, turn off the hot plate and allow the solution to cool down before taking the Al aliquot.

The sample is ready for the Al aliquot.

Al Aliquot

The objective of this procedure is to remove an aliquot of the sample to determine the total concentration of Al by AAS or ICP-MS.

Before you begin:

- **Label a narrow mouth Nalgene bottle of appropriate size (250 mL, 500 mL) with the weight of the bottle, the sample name, and 'HF' as a reminder that the bottle contains HF.**
- **Label a centrifuge tube on the top and side with the sample name, Al aliquot, weight of the tube, and 'HF' as a reminder that the centrifuge contains HF. If a centrifuge tube is not large enough, use a 60 mL narrow mouth Nalgene bottle.**
- **Use a blue Sharpie marker.**
- **Always wear a lab coat, acid coat, eye protection, face mask, and <u>two pairs</u> of nitrile gloves when handling HF!!**

1. In the HF fume hood: After the HF / HNO_3 solution has cooled down, transfer the solution from the Teflon beaker into the large labeled Nalgene bottle.

2. Weigh the bottle containing the solution and transfer 5% of the net solution weight into the labeled centrifuge tube. Record the weight of the solution and the aliquot on the worksheet. All transfers should be done in the HF fume hood. Note: Assume a density of 1 g/mL.

3. After the Al aliquot transfer is complete, pour the remaining solution back into the Teflon beaker. Rinse the Nalgene bottle three times with NP H_2O.

4. Place the Teflon beaker back onto a hot plate and continue heating the solution <u>uncovered</u> until all solution is completely evaporated and the sample is dry.

After the dry down, the sample is ready for perchloric fuming.

Perchloric Fuming

The objective of this procedure is to remove any trace amounts of fluoride (SiF_4) by evaporating off with $HClO_4$.

1. After the sample is completely dry, three $HClO_4$ fumings are performed.

2. In the $HClO_4$ fume hood: For the first fuming, add 10 mL of NP H_2O, 10 mL of HNO_3, and 4 mL of $HClO_4$ to the Teflon beaker. Place the beaker on a hot plate and heat to a sub-boiling temperature (hot plate setting of ~340ºC). Swirl the beaker around to dissolve the sample completely. Let the sample dry down completely before moving on to the second fuming.

3. In the $HClO_4$ fume hood: For the second fuming, rinse the Teflon beaker with NP H_2O (4 to 6 mL) and add 2 mL of $HClO_4$. Swirl the beaker around to dissolve the sample completely. Let the sample dry down completely before moving on to the third fuming.

4. In the $HClO_4$ fume hood: For the third fuming, rinse the Teflon beaker with NP H_2O (4 to 6 mL) and add 2mL of $HClO_4$. Swirl the beaker around to dissolve the sample completely. Let the sample dry down completely.

5. The $HClO_4$ fume hood must be rinsed down after each use! Set the timer on the left side of the fume hood to ~15 minutes to wash down the hood. Record your name and the date the fume hood was cleaned on the $HClO_4$ fume hood cleaning sheet located on the left side of the hood.

The sample is ready for ion exchange chromatography.

Ion Exchange Chromatography

The objective of this procedure is to isolate Be and Al by separating Fe and Ti from Be and Al from the sample.

Anion Column

Before you begin:

- **Label a centrifuge tube on the top and side with the sample name and 'AN' for anion.**
- **Label the following bottles:**
  - **60mL bottle with sample name, the column used, FRACTION 1, and 'AN'**
  - **125mL bottle with sample name, the column used, FRACTION 2, 'AN', and 'R' for reject**
- **When adding a solution to a column: Slowly, dispense a couple of milliliters of the solution by using a pipette to avoid disturbing the column resin. Then carefully pour the remainder of the solution into the column.**
- **Check off each fraction on the ion exchange column worksheet (Worksheet 2) as you go through the procedure.**
- **Keep all fractions except conditioning until the Be / Al analyses have been performed.**

1. In the $HClO_4$ fume hood: Add 3 mL of 9N HCl to the Teflon beaker containing the sample. Swish the beaker around and place the beaker on the hot plate to redissolve the sample. Once dissolved, transfer the solution into the labeled centrifuge tube. Then rinse the Teflon beaker with 2 mL of 9N HCl and transfer into the centrifuge tube. Rinse the beaker again with 1 mL of 9N HCl and transfer into the centrifuge tube. Make sure to use separate pipettes when measuring the HCl and when transferring the solution for each sample.

2. Vortex the centrifuge tube for ~15 seconds. After vortexing, centrifuge the tube for 3 minutes. Make sure to balance out the weight in the centrifuge.

3. Conditioning: Place a conditioning bottle under the column. Add 60 mL (3 cv) of 9N HCl to the column. Drain the column completely. The conditioning solutions can be discarded into the waste barrel. Note: 1 cv = 20 mL

4. Sample: Place the FRACTION 1 bottle under the column. Use a new pipette to transfer the entire sample into the column (use a new transfer pipette for each sample). Drain the sample through the column completely.

5. Fraction 1: Keep the FRACTION 1 bottle under the column. Add 40 mL (2 cv) of 9N HCl into the column. Drain the column completely.

6. Fraction 2: Place the FRACTION 2 bottle under the column. Add 80 mL (4 cv) of 0.1N HCl into the column. Drain the column completely.

7. After all fractions are complete, keep the columns moist by adding a few milliliters of 0.1N HCl. Anion columns should rest in weak acid.

8. Label a new Teflon beaker on both sides with the sample name and 'AN'.

9. In the HF or HNO_3 fume hood: Transfer the contents of the FRACTION 1 bottle into the labeled Teflon beaker. Rinse the bottle three times with NP H_2O. Place the Teflon beaker on a hot plate and heat to a sub-boiling temperature (hot plate setting of ~350°C) until the sample is completely dry.

Cation Column

Before you begin:

- **Label a centrifuge tube on the top and side with the sample name and 'CAT' for cation.**
- **Label the following bottles with the sample name, the column used, and 'CAT':**
  - **250mL bottle labeled FRACTION 1, 'R' for reject, 'Ti' for titanium**
  - **125mL bottle labeled FRACTION 2, 'Be' for Beryllium**
  - **125mL bottle labeled FRACTION 3, 'R' for reject**
  - **125mL bottle labeled FRACTION 4, 'Al' for Aluminum**
- **Check off each fraction on the ion exchange column worksheet (Worksheet 2) as you go through the procedure.**
- **Keep all fractions except conditioning until the Be / Al analyses have been performed.**

1. In the HF or HNO_3 fume hood: Add 3 mL of 1N H_2SO_4 and 1 drop of H_2O_2 to the Teflon beaker containing the dried sample. Swish the beaker around and place the beaker on a hot plate to redissolve the sample. Once dissolved, transfer the solution into the labeled centrifuge tube. Then rinse the Teflon beaker with 3 mL of 1N H_2SO_4 and transfer the solution into the centrifuge tube. Make sure to use separate pipettes when measuring the H_2SO_4 and when transferring the solution for each sample.

2. Vortex the centrifuge tube for ~15 seconds. After vortexing, centrifuge the tube for 3 minutes.

3. Sample: Place the FRACTION 1 bottle under the column. Use a new pipette to transfer the entire sample into the column (use a new transfer pipette for each sample). Drain the column completely.

4. Fraction 1: Keep the FRACTION 1 bottle under the column. Add 140 mL (7 cv) of 1N H_2SO_4 (0.1% H_2O_2) into the column. Drain the column completely.

5. Fraction 2: Place the FRACTION 2 bottle under the column. Add 100 mL (5 cv) of 1N HCl into the column. Drain the column completely.

6. Fraction 3: Place the FRACTION 3 bottle under the column. Add 120 mL (6 cv) of 1N HCl into the column. Drain the column completely.

7. Fraction 4: Place the FRACTION 4 bottle under the column. Add 80 mL (4 cv) of 2.5N HCl into the column. Drain the column completely.

8. Conditioning: Place a conditioning bottle under the column. Add 200 mL (10 cv) of 6N HCl into the column. Drain the column completely. Then add 60 mL (3 cv) of NP H_2O into the column. Drain the column completely. The conditioning solutions can be discarded into the waste barrel.

9. After all fractions are complete, keep the columns moist by adding a few milliliters of NP H_2O. Cation columns should rest in NP H_20.

10. Label a new Teflon beaker on both sides with the sample name and 'CAT'.

11. In the HF or HNO_3 fume hood: Transfer the contents of the FRACTION 2 (for Be) or the FRACTION 4 (for Al) bottle into the labeled Teflon beaker. Rinse the bottle three times with NP H_2O. Place the Teflon beaker on a hot plate and heat to a sub-boiling temperature (hot plate setting of ~350°C) until the sample is completely dry.

After the dry down, the sample is ready for hydroxide precipitation.

Hydroxide Precipitation

The objective of this procedure is to precipitate and wash the Be or Al that has been isolated from the sample.

Before you begin:

- **Label a centrifuge tube on the top and side with the sample name and 'Be' for Beryllium. Do the same for Al if you are measuring this nuclide.**

1. In the fume hood: Add 0.5 mL of 6N HCl to the Teflon beaker that contains the dried sample. Swish the beaker around and add another 0.5 mL of 6N HCl to the beaker. Once the sample is dissolved, transfer the solution into the labeled centrifuge tube. Make sure to use separate pipettes when measuring the HCl and when transferring the solution for each sample.

2. In the fume hood: Use a small amount of NP H_2O to rinse the beaker. Add enough NP H_2O to the centrifuge tube to get a total of 4 mL of solution in the tube.

3. Add 1 mL of 1:1 NH_4OH to the centrifuge tube. Vortex the tube for ~15 seconds and check for whitish particles. Add an additional 1 mL of NH_4OH if necessary and vortex again.

4. Let solution sit in the tube for ~30 minutes and then centrifuge the tube for 3 minutes.

5. Dump off the excess solution into the precipitant waste bottle. Then, add 3 mL of NP H_2O to the tube, vortex the tube for ~15 seconds, and centrifuge the tube for 3 minutes. Dump the off the excess solution into the precipitant waste bottle. Repeat with NP H_2O again and dump off excess solution.

The sample is ready for dry down and ignition.

Dry Down and Ignition

The objective of this procedure is to dry down and ignite the precipitated BeO or Al_2O_3 "gel".

Before you begin:

- **Make a "map" in your notebook of the placement of crucibles in the heat block.**
- **Make a "map" in your notebook of the placement of crucibles in the crucible rack for the furnace.**
- **Note: It is crucial that these maps be made and that samples be placed in the correct position in the heat blocks and crucible racks otherwise you will lose track of which samples are where. There is no other way to label samples at this stage of the procedure. If you screw this up, all of the work you have done up to this point will be lost!!!**
- **Label a glass vial on the lid and side with the sample name.**

1. Using a new pipette, transfer the contents of the centrifuge tube into a clean crucible. Try to not touch the sides of the crucible with the pipette. Rinse the centrifuge tube with a couple of drops of water and transfer into the crucible. Fill the crucible up with the sample until it is about 2/3 full. Make sure to use a separate pipette for each sample. Turn the heater on a low temperature setting of ~4 and let the sample dry down. Drying usually takes overnight.

2. After the sample is completely dry, place the crucibles into the crucible rack and cover each crucible with a clean lid. Place the crucible rack inside the furnace and turn on the furnace. Keep the crucibles in the oven at 750ºC for 5 minutes. Turn the furnace off and open the door to allow the crucible to cool down.

3. Once the crucible is cool, transfer the crucible into the labeled glass vial.

The sample is ready to be packed in the targets (cathodes).

Be Packing

The objective of this procedure is to pack the Be sample into the AMS cathode. The cathode is then shipped to LLNL-CAMS for analysis.

Before you begin:

- **Label a plastic vial on the lid and side with the sample name.**
- **Label a target (cathode) on the side with the sample name.**
- **Place aluminum foil or a clean Kimwipe in the bottom of the glove box.**
- **Clean the tweezers, curette, rod, and stainless steel target holder in the glove box with alcohol.**
- **Sand the rod on its side and top with the sandpaper and then clean again with alcohol.**

1. In the glove box: Place the labeled target (cathode) onto the stainless steel target holder. Remove the crucible from the glass vial with the tweezers. Add 1 full curette scoop of Nb powder into the crucible. First, use the rod to scrape down the sides of the crucible and then scrape the bottom of the crucible with the rod by using clockwise and counter-clockwise motions.

2. In the glove box: The sample is ready to be transferred into the target after all of the Be has been scraped off the crucible and the Nb and Be have been thoroughly mixed. Slowly, turn the crucible upside down over the target to allow the Be / Nb mixture to slide out of the crucible. Tap the target to with the tweezers to get the mixture to enter the target. The target should be nearly full. Place the rod in the target and tap with the hammer to pack the mixture. After packing the depth to the mixture should be as deep as the diameter of the hole is wide. Put the target into the labeled plastic vial.

3. In the glove box: Make sure to clean the tweezers, curette, road, and stainless steel target holder after each sample. Make sure to sand the rod and repeat cleaning after each sample. Use each side of the rod ~ 4 times before replacing.

Al Packing

The objective of this procedure is to pack the Al sample into the AMS cathode. The cathode is then shipped to LLNL-CAMS for analysis.

Before you begin:

- **Label a plastic vial on the lid and side with the sample name and Al for Aluminum.**
- **Label a target (cathode) on the side with the sample name and Al for Aluminum.**
- **Place aluminum foil or a clean Kimwipe in the bottom of the glove box.**
- **Clean the tweezers, curette, rod, and stainless steel target holder in the glove box with alcohol.**
- **Sand the rod on its side and top with the sandpaper and then clean again with alcohol.**

1. In the glove box: Place the labeled target (cathode) onto the stainless steel target holder. Remove the crucible from the glass vial with the tweezers. First, use the rod to scrape down the sides of the crucible and then scrape the bottom of the crucible with the rod by using clockwise and counter-clockwise motions. Approximate the amount of Al in the crucible and add the same amount of Ag powder into the crucible (ideally, you will want a 1:1 mixture of Al and Ag. Mix the Al and Ag thoroughly.

2. In the glove box: The sample is ready to be transferred into the target after the Al and Ag have been thoroughly mixed. Carefully, scoop out ~one curette of the Ag:Al mixture and transfer the mixture to the target. Tap the target to with the tweezers to get the mixture to enter the target. The target should be nearly full. Place the rod in the target and tap with the hammer to pack the mixture. After packing the depth to the mixture should be as deep as the diameter of the hole is wide. Put the target into the labeled plastic vial.

3. In the glove box: Make sure to clean the tweezers, curette, road, and stainless steel target holder after each sample. Make sure to sand the rod and repeat cleaning after each sample. Use each side of the rod ~ 4 times before replacing.

Other

Solution Recipes

Note: Always add water before acid.

0.1N HCl
952mL NP H_2O
8mL HCl

1N HCl
880mL NP H_2O
80mL HCl

2.5N HCl
759mL NP H_2O
200mL HCl

6N HCl
50% NP H_2O
50% HCl

9N HCl
250mL NP H_2O
750mL HCl

1N H_2SO_4 (0.01% H_2O_2)
698mL NP H_2O
20mL H_2SO_4
2mL H_2O_2

1:1 Nitric Acid Bath
50% NP H_2O
50% HNO_3

1:100 Nitric Acid Bath
100 parts NP H_2O to 1 part HNO_3

1:1 NH_4OH
50% NP H_2O
50% NH_4OH

1% HF / HNO_3 Ultrasonic Leaches

7.5 g/L (1st HF Leach)

Sample (g)	H_20 (mL)	HNO_3 (mL)	HF (mL)
30	3660	54	83
29	3538	52	80
28	3416	51	78
27	3294	49	75
26	3172	47	72
25	3050	45	69
24	2928	43	66
23	2806	42	64
22	2684	40	61
21	2562	38	58
20	2440	36	55
19	2318	34	53
18	2196	33	50
17	2074	31	47
16	1952	29	44
15	1830	27	42
14	1708	25	39
13	1586	23	36
12	1464	22	33
11	1342	20	30
10	1220	18	28
9	1098	16	25
8	976	14	22
7	854	13	19
6	732	11	17
5	610	9	14
4	488	7	11
3	366	5	8
2	244	4	6
1	122	2	3

15 g/L (2nd and 3rd HF Leaches)				15 g/L (2nd and 3rd HF Leaches)			
Sample (g)	**H_20 (mL)**	**HNO_3 (mL)**	**HF (mL)**	**Sample (g)**	**H_2O (mL)**	**HNO_3 (mL)**	**HF (mL)**
60	3660	54	83	30	1830	27	42
59	3599	53	82	29	1769	26	40
58	3538	52	80	28	1708	25	39
57	3477	51	79	27	1647	24	37
56	3416	51	78	26	1586	23	36
55	3355	50	76	25	1525	23	35
54	3294	49	75	24	1464	22	33
53	3233	48	73	23	1403	21	32
52	3172	47	72	22	1342	20	30
51	3111	46	71	21	1281	19	29
50	3050	45	69	20	1220	18	28
49	2989	44	68	19	1159	17	26
48	2928	43	66	18	1098	16	25
47	2867	42	65	17	1037	15	24
46	2806	42	64	16	976	14	22
45	2745	41	62	15	915	14	21
44	2684	40	61	14	854	13	19
43	2623	39	60	13	793	12	18
42	2562	38	58	12	732	11	17
41	2501	37	57	11	671	10	15
40	2440	36	55	10	610	9	14
39	2379	35	54	9	549	8	12
38	2318	34	53	8	488	7	11
37	2257	33	51	7	427	6	10
36	2196	33	50	6	366	5	8
35	2135	32	48	5	305	5	7
34	2074	31	47	4	244	4	6
33	2013	30	46	3	183	3	4
32	1952	29	44	2	122	2	3
31	1891	28	43	1	61	1	1

Cleaning Procedures

Teflon and Glass
All Teflon and glass should be washed with detergent, scrubbed, and rinsed three times with DI H_2O. Then the Teflon and glass should be put in a heated 1:1 HNO_3/H_2O bath for 4 to 8 hours. Make sure to put the immersion heater in a glass beaker away from the Teflon beakers and the sides of the bath container. The immersion heater should be set on ~4.5.

Plastic
All plastic should be washed with detergent and rinsed three times with DI H_2O. Then put the plastic into an overnight 1:100 HNO_3/H_2O bath.

Large Glass Beakers (1 L/4 L)
All large glass beakers should be washed with detergent, scrubbed, and rinsed with DI H_2O.

4L HDPE Canisters
All canisters should be rinsed with DI H_2O, then wiped with a Kimwipe, and rinsed with DI H_2O again.

Packing the Ion Exchange Columns

The columns should be repacked after every five to ten samples or if samples from a completely new location are being processed.

Fill a 250 mL glass beaker with resin up to ~100 mL. Use a scoopula to transfer the resin into the beaker. Add ~100 mL of NP H_2O to the beaker. Stir the resin and NP H_2O with a pipette. Let the solution sit until the resin has settled into the bottom of the beaker. Use the pipette to transfer the resin into the columns. Fill the columns slowly to avoid trapped air bubbles. To remove a trapped air bubble, poke the resin with the pipette and tap the side of the column. You may need to drain off the excess NP H_2O as you are filling the columns. Fill the columns up to the 20 mL mark with the resin.

This procedure is the same for both the cation and anion exchange resin.

Quartz Crucible Cleaning

New Crucibles and Lids

1. Place ~100 new (do not use old ones!) crucibles in a 500 mL Teflon beaker. Crucibles should make a layer at the bottom of the beaker only two to four layers thick.

2. Fill the Teflon beaker with a 1:1 HNO_3 and 10% HF (115 mL HNO_3 and 35 mL HF). Add enough solution to cover the crucibles by one or two centimeters.

3. Put a Teflon lid on the beaker and boil for one hour.

4. Cool, drain acid to waste and rinse 3 times with NP H_2O.

5. Dry in oven in glass plate or glass beaker.

6. Place dry crucibles in closed box in the drawers beneath the furnace.

7. Write down on the log sheet, your name, date, and number of crucibles cleaned.

Used Lids

1. Place lids in a 500 mL Teflon beaker.

2. Fill beaker with 1:1 HNO_3 and 2.5% HF (141 mL HNO_3 and 9 mL HF). Add enough solution to cover the lids by one or two centimeters.

3. Put a Teflon lid on the beaker and boil for one hour.

4. Cool, drain acid to waste and rinse 3 times with NP H_2O.

5. Dry in oven in glass plate or glass beaker.

6. Discard any lids with holes and place remaining lids in the closed box in the drawer beneath the furnace.

7. Write down on the log sheet, your name, date, and number of lids cleaned.

<u>Setting Timers</u>

Setting the countdown timer

1. Display CD PROG by using the < and > key.

2. Press the SET key. The word SET will light up and the word ON will be flashing.

3. Select ON using the up/down key. Press the SET key.

4. The field of Hour should be flashing. Adjust the hour to desired time by using the < or > key. Press the SET key.

5. The field of Minute should be flashing. Adjust the minute to desired time by using the < or > key. Press the SET key.

6. The field of Second should be flashing. Adjust the second to desired time by using the < or > key. Press the SET key.

7. Press the SET key to complete the setting of the countdown timer.

8. To activate the timer, press the CD key. To stop the countdown, press the CD key again and the display will return to its pre-set countdown value.

9. When the countdown timer is operating, the word CD will be flashing on the display.

Barnstead NanoPure H_2O Unit

Setting Volumetric Dispensing

1. From the normal recirculating mode, when the display is showing a purity of 18.2 MΩ-cm, press the UP/DOWN arrow until the display reads, "Auto Dispense Menu?"

2. Press ENTER.

3. Display will read, "Dispense Method." Press the UP/DOWN arrow to select "VOLUMETRIC."

4. Press ENTER. Display will read "Unit Volume Setting: xx Liters."

5. Press the up/down arrow to set the desired volume value. Press ENTER.

6. Press the Dispense key to dispense the NP H_2O. Make sure the tube is connected to the Barnstead unit and in the carboy before dispensing!

System Cleaning

Note: The cleaning timer will display after six months, reminding you to clean the unit. Complete the system cleaning and reset the cleaning timer. It is best to perform cleaning procedures early in the afternoon. After cleaning is complete (~3.5 hours), place the unit into its normal recirculation mode to allow the system to recirculate overnight after cleaning.

1. From the normal recirculating mode, press the START button once to put in Idle mode. Once in Idle mode, press the UP/DOWN arrow until the display reads "Clean Unit?"

2. Press ENTER.

3. Display will prompt, "Disconnected Inlet Water (NO)."

4. Disconnect the feed water supply at the quick disconnect inlet valve located on the left side of the unit.

5. Press the UP/DOWN arrow to select (YES) and then press ENTER. Display will now prompt, “Injected Cleaner (NO)?”

6. Remove solution syringe from wrapper. Open front door. Remove luer cap on system injection port, located on the right side of the cartridge pack manifold by turning counterclockwise.

7. Remove the syringe luer cap and attach the syringe to the system luer fitting. Slowly, inject the solution into the system and remove syringe. Avoid injecting air into the system. Replace luer cap on injection port. Close door.

8. Press the UP/DOWN arrow to select (YES) and then press ENTER. Display will now read “Reconnected Water (NO).”

9. Attach feed water supply removed in Step 4.

10. Press the UP/DOWN arrow to select (YES) and press ENTER. Display will now read, “Unit Cleaning.”

11. Unit may be left unattended until the display reads, “Cleaning Complete: Press ENTER.” After cleaning is complete, and the user presses “ENTER” the unit will return to the Idle mode. Press the START button to return the unit to the normal recirculating mode.

Resetting the Cleaning Timer

1. From the Idle mode, when the display reads “NANOpure Diamond (Idle) xx:xx:xx xx/xx/xx,”, press the UP/DOWN arrow until the display reads, “Reset Timers?”

2. Press ENTER.

3. Display will read, “Reset Cleaning Timer” and show on the right side of the second line when the user will start receiving “Clean Unit” reminders.

4. Press the UP/DOWN arrow to select “YES.”

5. Press ENTER and the timer will be reset. This will reset the cleaning timer for ~ 6 months.

Chematix

Chematix is the chemical management system that Georgia Tech uses to inventory chemicals. You may log into Chematix using your GT Account and Password at the following site:

https://www.chematix.gatech.edu/Chematix/

Adding Chemicals to Inventory

1. Under the inventory tab, located at the top of the page, click on "add chemical container(s) to your inventory".

2. Select the chemical that you are adding or search for the chemical.

3. Once a chemical is selected, enter the size of the container, concentration, and lab location. Click on submit. You will be notified to apply the barcode to the container.

Removing Inventory

1. Under the inventory tab, located at the top of the page, click on "reconcile storage unit inventory".

2. Choose your location and click on reconcile storage unit.

3. Select the chemical(s) you would like to remove. There are several options for removing chemicals. The options include: (1) click on "mark as consumed" if the chemical has been consumed or (2) designate the chemical as "discarded as solid waste" or "discarded as liquid waste" and then click on "mark as discarded".

Emergency Procedures

Report all accidents and emergencies to laboratory director.

Serious accidents should be reported to the laboratory director, Georgia Tech Environmental Health and Safety (http://safety.gatech.edu), and Georgia Tech Police (404-894-2500) IMMEDIATELY. For serious emergencies DO NOT call 911. Emergency phone numbers are located by the doors of all laboratory rooms.

In case of fire, leave laboratory, shut door, pull fire alarm, and call Georgia Tech police (404-894-2500).

If an acid spill occurs, use designated spill kits (located under the sinks on both sides of the laboratory) and procedures.

For minor burns or other non-chemical injuries (cuts, scrapes, etc.), use the first aid kit. If acid comes in direct contact with clothing, immediately remove affected clothing.

If acid comes in contact with skin, rinse the contacted area thoroughly the sink or emergency shower. If necessary, seek further medical treatment.

For contact with HF, apply calcium gluconate gel (located above ultrasonic tanks and on side of HF fume hood) after brief rinsing with water. If significant exposure to HF occurs, rinse with water, apply calcium gluconate gel and seek immediate emergency medical attention.

In the case of contact with acid in eyes, rinse eyes thoroughly (keep non-affected eye closed) at eye wash stations for 15 minutes and proceed to hospital.

If hazardous fumes are inhaled, immediately get fresh air.

Binders containing the MSDS for all chemicals in the lab are located near each sink.

Most importantly, use common sense!

Worksheet 1

1 Sieved rock 250-500µm

2 HCl leach — 6N HCl + 0.03% H_2O_2, 10ml/g; 12 hours sub-boiling

3 HF leach — 1%HF + 1% HNO_3

	HF	HNO_3	H_2O	Canister x amount	Initial	Final
1. 7.5g/L; 9hours at 90°C						
2. 15g/L; 9hours at 90°C						
3. 15g/L; 9hours at 90°C						

4 Be + Al Carrier — Teflon beaker + quartz + Be carrier (0.3mg) + Al carrier (2mg)

quartz (g)		
Be carrier (mg)	concentration	name
Al carrier (mg)	concentration	name

5 $HF + HNO_3$ — HF (5ml/g) + HNO_3 (0.3ml/g)

HF	HNO_3

lid on beaker; heat 1-2hrs until completely dissolved; cool down; transfer to tared bottle

6 Al aliquot — weigh solution + take aliquot

solution (g)	aliquot (g)

dry down completely

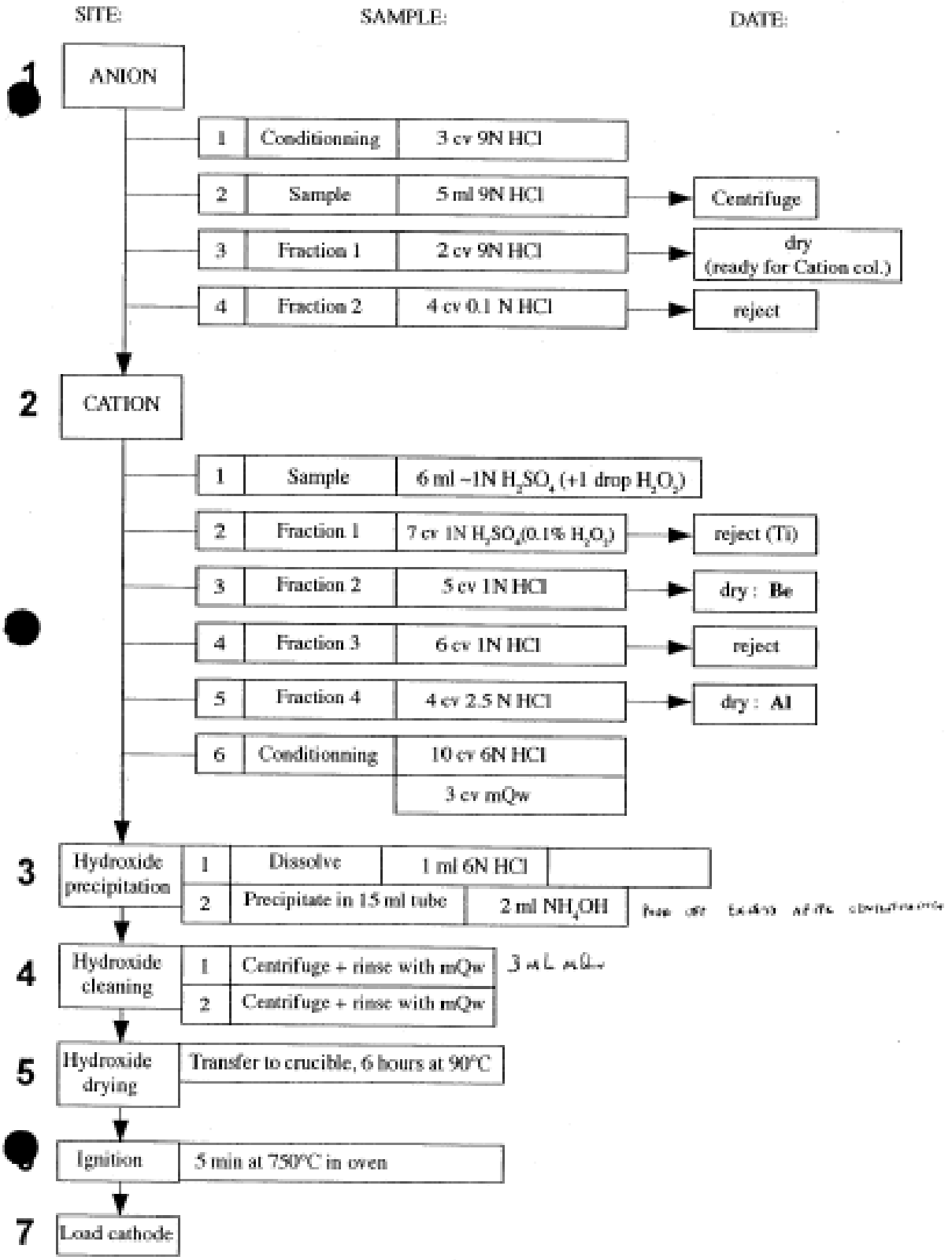
7 $HClO_4$ fuming

1. 10mL H_2O + 10mL HNO_3 + 4mL $HClO_4$
2. H2O rinse + 2mL HClO4
3. H_2O rinse + 2mL $HClO_4$

dry down completely between fuming steps

dry (ready for columns)

Worksheet 2


SITE:
SAMPLE:
DATE:
1
ANION
1 Conditionning 3 cv 9N HCl
2 Sample 5 ml 9N HCl
Centrifuge
3 Fraction 1 2 cv 9N HCl
dry (ready for Cation col.)
4 Fraction 2 4 cv 0.1 N HCl
reject
2
CATION
1 Sample 6 ml ~1N H_2SO_4 (+1 drop H_2O_2)
2 Fraction 1 7 cv 1N H_2SO_4(0.1% H_2O_2)
reject (Ti)
3 Fraction 2 5 cv 1N HCl
dry : Be
4 Fraction 3 6 cv 1N HCl
reject
5 Fraction 4 4 cv 2.5 N HCl
dry : Al
6 Conditionning 10 cv 6N HCl
3 cv mQw
3
Hydroxide precipitation
1 Dissolve 1 ml 6N HCl
2 Precipitate in 15 ml tube 2 ml NH_4OH
4
Hydroxide cleaning
1 Centrifuge + rinse with mQw
2 Centrifuge + rinse with mQw
5
Hydroxide drying
Transfer to crucible, 6 hours at 90°C
Ignition
5 min at 750°C in oven
7
Load cathode