

UNIVERSITY OF PENNSYLVANIA LABORATORY PROCEDURES FOR PREPARATION OF Be AND Al SAMPLES

I. PHYSICAL PRETREATMENT

A. Sample crushing and coarse grinding (in Geology Department)

1. Rock sample is crushed in jaw crusher, and ground using plate grinder, being careful not to grind the sample too fine.
2. Sample is sieved through 420 μ sieve. Plate grinder is readjusted and new material >420 μ is not through grinder and sieging again. This continues

$\leq 420\mu$.

B Fine Grinding (in DRL)

1. Sample is weighed.
2. Sample is sieved through 150 and 250 μ sieves.
3. Small quantities of rock material >250 μ (just enough to cover SiC balls in grinding chamber) are ground in Spex Wig-L-Bug mill for short periods of time (1 to 2 minutes depending on individual rock sample)

II. CHEMICAL PRETREATMENT - Deionized water and Teflon digestion vessels are used throughout procedures that follow. (Whenever something is weighed, the weight is recorded.)

A. Aqua Regia.

- 1. If 150 to 250 μ fraction is sufficient in size it is boiled in Aqua Regia for 2 hours. (If not large enough, combine with 75-150 μ fraction and boil in Aqua Regia.)**
- 2. Cool sample and allow to settle. Decant and save supernate (ALL SUPERNATES ARE LABELED AND SAVED).**

2. Heavy fraction is removed through stopcock of separatory funnel onto filter paper in regular funnel. This and all subsequently removed fractions are washed with acetone repeatedly to remove bromoform (bromoform/acetone mixture is saved for recycling). This and all subsequently separated mineral fractions are air dried (covered with filter paper) and weighed.

7. Residual bromoform is removed from the ...

3. Plagioclase (> 3% An) is stained pink to red.

4. "Q" remains unstained, but may appear more or less frosted.

~~_____ stained and _____ unreactive sample can be cut back~~

through ~~transform~~ for further separation if necessary.

V. CHEMICAL PREPARATION OF Al AND Be SAMPLES

Deionized water and Teflon digestion vessels are used throughout procedures that follow.

(When sample weight is determined, acid weight is recorded.)

d. Add 20 ml conc. HF and dissolve sample on 100 C hotplate for a

e. Add 10 ml conc. HCl and evaporate to dryness (to get rid of HF).

f. Repeat step e.

g. Dissolve dried sample in about 20-30 ml 0.5N HCl. Centrifuge and decant if there is any undissolved material remaining. Pour supernate into weighed 100 ml volumetric flask. Wash residue 2 to 3 times with 0.5N HCl centrifuge and decant into volumetric. Make volumetric up to 100 ml with 0.5N HCl and weigh on analytical balance.

h. Pipette 5 ml of sample solution into each of 2 previously weighed 10 ml volumetric flasks labeled A & B.

i. Weigh 100 ml volumetric flask after removing ICP samples. Add 1 ml Al carrier and weigh again.

j. Weigh ICP sample added to each 10 ml volumetric flask. To B add 1 ml of Al "AA" solution with pipette and weigh flask again. Make each

m. Add H_2O to precipitate in centrifuge tube to bring up to 5 ml and

with 2.5% HF and shake or vortex. Allow to stand briefly and adjust pH if necessary. Add 1:1 NH_4OH to bring pH back up to 8. Centrifuge and decant supernate into small digestion vessel. This is the Be fraction. The precipitate (Al sample) remaining in 15 ml tube is put aside until Be is finished being processed.

2. Be FRACTION.

a. Be supernate is evaporated to dryness on 200 C hotplate. Dried sample is then re-dissolved in 10ml 2.5% HF and pH adjusted once again to 8 with 1:1 NH_4OH and centrifuged. The Be Solution is decanted back into the small teflon digestion vessel and once again

step is saved to be combined with Al portion previously saved.

3. Al FRACTION

- a. Dissolve Al precipitate in 10 ml conc. HCl and transfer to hotplate.
- b. Add 10 ml conc. HCl and evaporate to dryness (to get rid of HF).
- c. Repeat step b.
- d. Dissolve in 10 ml 0.5N HCl/2% NH₄Cl solution. Heat. Adjust pH to

save supernate as supernate #3.

supernate #3. Repeat washing once again.

- f. Transfer Al(OH)₃ sample to quartz crucible by pipetting in a similar fashion to that used for Be.

- g. Dry in oven at 110 C to remove H₂O.