TO: PAUL BIERMAN

FROM: MILAN PAVICH

SUBJECT: GARDEN VARIETY ^{10}Be

HERE'S THE DIGESTION METHOD FIRST DEVELOPED BY FOUAD TERA (DTM) AND MODIFIED BY SHUHUI ZHENG.

I USED IT FOR SEVERAL YEARS IN MY LAB.

SHUHUI NOW USES A COLUMN METHOD, I BELIEVE. YOU MAY BE ABLE TO REACH HER AT UC-IRVINE (szheng@uci.edu).

CHEERS,

Milan.

^{10}Be Chemistry for Sediments

Before extraction:

- Mechanical clean the used teflon jar; soak in 1:1 HCl for several days; rinse by distilled water; oven dry at 60°C.
- Mechanical clean the used alumina crucible; soak in conc.HNO_3 for several days on hot plate (~ 100°C); rinse by water; oven dry.
- Rinse 50ml and 15ml screw-top vials with 1:1 HCl; rinse by water; oven dry at 60°C.
- Grind sediments sample to fine powder; oven dry at 55-60°C for 24hrs, store in vials in the dessicator.

1) Sample decomposition.

- Weigh samples; for surface samples (~10^9atoms/g), the amount is ~0.2g; down to the cores, from 0.5g to 1g; place the sample in 60ml teflon jar.
- Weigh out ^{9}Be spike into jar; weight of spike is 0.5-1mg ^{9}Be.
- Wash down sides of jar with ~5ml water.
- add 5-10ml HF(49%) and 2ml $HClO_4$(72%)
- Let sit with cape on, but loose for several hrs.
- Dry down samples on hot plate ~200°C, sometimes add more $HClO_4$ to further oxidize the black organic stuff in the jar.
- Dry down hard until sample forms solid cake.
- Add 10ml 1:1HCl, swirl well, be sure all sample taken up, then dry down again.

2) Precipitation of $Fe(OH)_3$ with Be & Al etc.

- Take up sample in 20ml 3N HCl; let sit until it forms clear solution and transfer to 50ml vial.
- add 15ml conc. NH_4OH, pH~8; shake well; sit overnight to complete the precipitation of hydroxides.

3) Separation of Be from Fe and Al.

- Centrifuge; decant the supernate; wash the precipitation twice with conc.NH_4OH; using 3:1 NH_4OH/ppt.ratio.
- Using Vortexter to homogenize ppt. and NH_4OH.
- Centrifuge 10-15min ; decant supernate as quantitatively as possible.
- Add 1:10 HF to residue in 2:1 HF/residue ratio.
- Using Votexter to homogenize.
- Add more 1:10 HF until the solution turns to ivory (pH~5).
- Add conc.NH_4OH dropwise until the solution turns to brown again, pH~7; wait for 1 hr to let complete precipitation of $Fe(OH)_3$.
- Centrifuge; pour supernate (Be) into original jar(after washed with 3N HCl, then rinse with water); discard the residue with the vial.

4) Final product - BeO

- Dry down the solution in the jar on the hot plate overnight at 200-250°C, (under Al foil canopy)

- mixed up ~1ml 20% $HClO_4$ to cover the sample; dry down at ~200°C.
- Repeat the above proceedure.
- Take up sample in 1ml 1:1 HCl; dry down at ~150°C.
- Repeat the above proceedure.
- Take sample up in 10ml 1NHCl and transfer the solution to 15ml vial.
- add ~40 drops of conc. NH_4OH; shake well; stay for 1hr.
- Centrifuge; decant the supernate.
- Transfer the gel $Be(OH)_2$ to alumina crucible.
- Bake the crucible in furnace; 2hrs at 80°C, 1hr at 550°C.

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