## Cleaning the sample turret

1. Disassemble turret: Screws, springs, ball-bearings, retaining ring and screw are not cleaned (store in small beaker); blocks and turret go in one beaker and hats in another beaker and both go into clean lab.
2. Add reagent grade 4M HNO3 to both beakers (enough to cover plus 2”) and boil (ceramic hot plate at 250°C) for 30 minutes in the clean lab; cool, and rinse 2x with MQH2O.
3. Add ACS-grade hydrogen peroxide (H2O2) to both beakers (enough to cover plus 2”) and boil (hot plate at 180°C) for 30 minutes on the ceramic hotplates in the clean lab **– make sure the parts stay submerged in peroxide.** Let cooland rinse 2x with MQH2O.
4. Dry any water on the outside and edges of beaker with a wiper, then rinse the parts with methanol and dry in the 50°C oven in the mass spec lab; when dry transfer to dry box.

## Cleaning filament beads

1. Gently strip the old Re filament material from the posts with tweezers and place in a small clean Pyrex beaker.
2. In the clean lab, generously immerse the filaments in ACS-grade hydrogen peroxide (H2O2), place on the ceramic hotplates in the clean lab and boil for 30 minutes **– make sure the filaments stay submerged in peroxide.** Let cool and rinse 2x with MQH2O.
3. Immerse the filaments in Omnisolv methanol, and again boil on the ceramic hotplates in the clean lab for 30 minutes - **again make sure the filaments stay immersed in methanol.**
4. Cool and rinse with fresh methanol or isopropanol, dry the filaments in the 50°C oven in the mass spec lab; when dry label beaker and transfer to dry box.

## Cleaning the ion collimator

1. Disassemble ion collimator stack; remove the grub screws; be very attentive as to how the collimator comes apart for when you have to reassemble… digital photos or diagrams help. Ceramics and screw are not generally cleaned (store in small beaker); neither is the large chunk of stainless steel nor the last accordion piece.
2. If necessary, place the ceramic parts in a small beaker with reagent grade hydrogen peroxide (H2O2) (enough to cover plus 2”) and boil (hot plate at 180°C) for 30 minutes on the ceramic hotplates in the clean lab hood; cool, and rinse 2x with MQH2O. Skip to step 5.
3. Place the stainless steel lens plates and slits in a small beaker with reagent grade 4M HNO3 to both beakers (enough to cover plus 2”) and boil (hot plate at 250°C) on the ceramic hotplates in the clean lab hood; cool, and rinse 2x with MQH2O.
4. Add ACS-grade hydrogen peroxide (H2O2) (enough to cover plus 2”) and boil (hot plate at 180°C) for 30 minutes on the ceramic hotplates in the clean lab hood; cool, and rinse 2x with MQH2O.
5. Do a final rinse on all parts with isopropanol or methanol and dry in the 50°C oven in the mass spec lab; when dry transfer to dry box.

## Welding filaments

Re filament material is welded between the two stainless steel posts of the filament assembly by electrical capacitance discharge at 10A.

With the filament assembly situated sideways relative to the welder, the Re should be welded to the outer side of the far post with 2-3 welds, stretched taut and flat across the posts and welded to the outer side of the near post with 2-3 welds.

The excess Re ribbon can then be cut with a scissors from the filament assembly and the “tail” welded to the post.

Care should be taken never to handle the filaments without gloves, or to touch the filament once welded to the posts.

## Outgassing filaments

The strung filaments are placed in the outgasser, pumped until a vacuum of < 1e-7 torr is achieved.

Turn on the chiller.

Outgas filaments according to the following schedule: 15 minutes at 2A, 30 minutes at 4A (zone-refined filaments for U metal analysis should be outgassed at 4A for 60 minutes).

Allow filaments to cool for ~15 minutes, then vent the outgasser to dry nitrogen, and removed the filaments and store in the (left-hand) Secador dry box.

Allow the filaments to sit at atmosphere AT LEAST overnight before attempting to load samples—solutions may spread across filament otherwise.

## Silica gel emitter preparation

Procedure modified from Gerstenburger and Haase, 1997, Chem. Geol.

#### Materials

0.2 g colloidal silicic acid (Merck)

19.6 g 0.035M H3PO4 (diluted from concentrated orthophosphoric acid)

#### Method

Using an acid-washed 250µl pipet tip and a clean pipettor, draw approximately 200 µl of colloidal silicic acid from the very top of the undisturbed 30 ml bottle of stock solution, and weigh into a tared, empty, clean 7 ml Savillex beaker.

Dilute the colloidal silicic acid with 0.035M H3PO4 (make sure the molarity of the dilute phosphoric acid is accurate by titration) in the weight ratio, 0.2 g silicic acid : 19.6 g 0.035M H3PO4. To do so, weigh the appropriate amount of 0.035M H3PO4 into a 30ml spaghetti tube-tipped dropper bottle, and place it in an ultrasonic bath. With your clean pipet tip, pipet the weighed colloidal silicic acid from your beaker into the bottle dropwise, allowing the silicic to disperse under sonication.

The resulting dilute silica gel emitter should yield loading blanks less than 200 fg Pb, and ion currents of greater than 10,000 cps per pg (measured on 205Pb spiked loading blanks).

It is recommended to sonicate the bottle of silica gel solution regularly to maintain the dispersed silica in solution.

If the silica gel solution yields anomalously high blanks, then it can be cleaned by treatment with coarse anion exchange resin in a dilute HBr medium as follows:

1. To the ~20 ml of silica gel solution mixed above, add 1.11 mL (1.65 g) of conc. (9M HBr) to yield a 0.5M HBr solution.
2. Starting with dry AG1-X8 20-50 mesh resin, immerse a batch in MQH2O in a small bottle (e.g. 250 mL), shake and let the coarsest resin settle before decanting the fines. Repeat the filling, shaking and decanting of the fines until you have a well-sized coarse resin fraction.
3. Add enough clean 6M HCl to the bottle to cover the resin, let equilibrate for an hour, the decant the 6M HCl. Repeat this process several times, alternating MQH2O and 6M HCl. Finish this procedure with several rinses of MQH2O prior to storing the resin in MQH2O.
4. Add approximately 2 mL of wet, sized, clean resin to the silica gel mixture in 0.5M HBr and recap. Shake this bottle occasionally over the course of a day and let sit overnight, or place in a automatic shaker for 24 hours.
5. The next day, decant the silica-gel mixture into a new dropper bottle, being careful not to transfer any resin to the new bottle. The cleaned silica gel should have loading blanks reduced by at least 50% compared to the starting composition.

## Tantalum oxide emitter solution preparation (traditional)

#### Materials

2 g TaCl5 (Aldrich 99.9% or equivalent)

1.2 ml concentrated HF (50%)

1.2 ml concentrated H3PO4 (85%)

20 ml concentrated HNO3 (70%)

80 ml MQH2O

#### Method

* + 1. Weigh the 2 g of TaCl5 into an empty, dry and clean 125 ml FEP bottle.
    2. Add 30 ml of MQH2O; TaCl5 reacts with water and is hydrolyzed to a white solid (Ta2O5).
    3. Add HF, H3PO4, and HNO3; the white solid in suspension should dissolve rapidly or at least within a couple of hours.
    4. Dilute with the remaining MQH2O.

If the loading blank isn’t good enough, Ta can be purified by the following method:

1. After the hydrolysis, and before adding the HF), centrifuge the white precipitate and discard the supernatant.
2. Wash the powder several times with 3M HCl and/or 4M HNO3; no Ta2O5 should dissolve.
3. Proceed with addition of the HF, H3PO4, and HNO3, and dilution.

**“Tantalum Gel” for Rb & Sr analysis (Rob Creaser recipe)**

(Also works brilliantly for Sm as Sm+ and Nd as NdO+ off single Re)

**Manufacture:**

Obtain good quality TaCl5 (I use Strem Chemicals, resublimed 99.99% TaCl5, cat#93-7324, <http://www.strem.com/code/index.ghc>)

Dissolve 3.0 grams TaCl5 in a mix of 25 ml 95% ethanol and 25 ml MilliQ water (or equivalent

~ 18.2MΩ water) – it should dissolve rapidly (minutes).

At this point I run the dissolved Ta solution through a cation column (~ 4ml resin bed volume, equilibrated with MilliQ) to remove any Rb and Sr contaminant, collecting the Ta solution in a cleaned Teflon screw-cap jar (Ta does not bind to the cation column).

Put the lid on the jar of Ta solution only a half turn or so to allow the EtOH to slowly evaporate, and in ~ 2-5 days the remaining Ta will have turned to gel. Don’t ask me exactly what happens here.

For a working solution take equal volumes of Ta Gel and MilliQ water into a small bottle (e.g.,

3 mls of each). You may need to ultrasonicate it every month or so to stop it “clumping”. Keep the stock solution tightly capped - my current batch has been fine for 4 years on a shelf.

**Loading:**

Load Rb or Sr to single Re filament in MilliQ water and dry at ~ 1 A

For each sample, add 3.5 µl Ta gel and 1.5 µl ~ 1 N H3PO4 into a drop on parafilm, then add all 5 µl on top of the loaded Sr or Rb on the filament.

Slowly turn up the filament as the solution evaporates, but not so fast that it bubbles.

At ~ 2.2A the H3PO4 will fume off, and the load will “crystallize”.

I turn the filament up to dull red glow for 1-2 seconds to remove organics, then shut off. The load should look white and crystalline – it is quite robust.

Rb runs ~ 1.5 A and Sr at ~ 2.8A using 0.0012” thickness Re ribbon.

Standard Rb yields 87/85 of 0.3818 (much lower than NBS984 value) but is quite consistent.

5 ng of Sr should easily yield > 1 volt (1e-11Ω input resistor) 88Sr for > 30 minutes. R.A.Creaser

January 2006

## Loading filaments

#### Pb/UO2:

Outgas Re center filaments at 4A for 60 minutes.

Squeeze a microrop of silica gel-H3PO4 mixture into the cap of the sample beaker.

With a finger pipet, add 1-2 µl of silica gel-H3PO4 mixture to sample in beaker.

Let sit for a minute for sample to dissolve in silica gel.

Load full sample with finger pipet and spaghetti tubing onto Re filament and allow to air-dry flat on the filament. Once sample is evaporated, take filament current up at a rate of ~0.2A per second to just over 2.5A, or when filament just starts to glow. Immediately turn off the current.

Sample will turn gray to black at around 2.2A and may briefly fume at about 2.5A while turning light grey. Do not allow the sample to turn very white, as the load can then become flaky and fall off with subsequent handling.

#### U/Th:

Outgas zone refined Re center filaments at 5A for 60 minutes.

Squeeze a microrop of 3M HCl into the cap of the sample beaker.

With a finger pipet, add 1-2 µl of 3M HCl to sample in beaker.

Load 3 µl of dilute colloidal graphite solution (7 parts MQH2O to 1 part Ted Pella graphite) to the middle of an outgassed zone-refined Re center filament.

Load sample onto top of graphite solution, and allow to air-dry flat on the filament. Once sample is evaporated, take filament current up at a rate of ~0.2A per second to just over 2.5A, or when filament just starts to glow. Immediately turn off the current.

#### Sr (Creaser Ta gel):

Outgas Re center filaments at 4A for 60 minutes.

With the 0.5-10 µl pipet and a clean tip, add 4 µl 1M HNO3 to samples in beakers; let sit for a few minutes in order for samples to dissolve.

Sonicate and shake the 30 ml dropper bottle of “Creaser Ta Gel” for at least 5 minutes.

In the cap of the beaker, mix 3.5 µl of Creaser Ta Gel to 1.5 µl of 1N H3PO4.

Load 2 to 4 µl (half to all) of each sample with pipet and clean tip onto middle of outgassed Re center filaments (loader will take up to six filaments); take current up to 1A and allow to dry flat.

Load Ta gel – H3PO4 mixture onto sample spot and allow to air-dry flat on the filament. Once sample is evaporated, take filament current up at a rate of ~0.2A per second to just over 2.5A, or when filament just starts to glow. Immediately turn off the current.

#### Sr (Birck Ta slurry):

Outgas Re center filaments at 4A for 60 minutes.

Sonicate and shake the 30 ml bottle of “Birck” Ta slurry emitter solution for at least 5 minutes.

With the 0.5-10 µl pipet and a clean tip, add 4 µl 1M HNO3 to sample in beaker; let sit for a few minutes in order for sample to dissolve.

Load 4 µl of “Birck” Ta slurry emitter solution with pipet and clean tip onto middle of outgassed Re center filament and allow to air dry flat.

Load 2 µl of sample (that is half the solution) with pipet and clean tip on top of the dried Ta bed and allow to air dry flat on the filament. Once sample is evaporated, take filament current up at a rate of ~0.2A per second to just over 2.5A, or when filament just starts to glow. Immediately turn off current.

#### Rb (Creaser Ta gel):

Outgas Re center filaments at 4A for 60 minutes.

With the 0.5-10 µl pipet and a clean tip, add enough 1M HNO3 to sample in beaker to be able to easily extract the equivalent of ~50 ng of Rb; for example, if you estimate that you have 500 ng of Rb in the beaker, then adding 10 µl of 1M HNO3 will let you load 50 ng of Rb in a 1 µl aliquot of that solution. Let sit for a few minutes in order for sample to partially dissolve.

Sonicate and shake the 30 ml dropper bottle of “Creaser Ta Gel” for at least 5 minutes.

In the cap of the beaker, mix 3.5 µl of Creaser Ta Gel to 1.5 µl of 1N H3PO4.

Load enough sample solution to deliver ~50 ng of Rb (see above) with pipet and clean tip onto middle of outgassed Re center filaments (loader will take up to six filaments); take current up to 1A and allow to dry flat on the filament..

Load Ta gel – H3PO4 mixture onto sample spot and allow to air-dry flat on the filament. Once sample is evaporated, take filament current up at a rate of ~0.2A per second to just over 2.5A, or when filament just starts to glow. Immediately turn off current.

#### Nd:

Outgas Re center and side filaments at 4A for 60 minutes.

With the 0.5-10 µl pipet and a clean tip, add 4 µl 1M HNO3 to sample in beaker; let sit for a few minutes in order for sample to dissolve.

Load 2 to 4 µl (half to all) of sample with pipet and clean tip onto middle of one outgassed Re side filament, take current up to 1A and allow to dry flat on the filament.

Once sample is evaporated, take filament current up at a rate of ~0.2A per second to just over 2.5A, or when filament just starts to glow. Immediately turn off current.

#### Sm (Creaser Ta gel):

Outgas Re center filaments at 4A for 60 minutes.

With the 0.5-10 µl pipet and a clean tip, add 2 µl 1M HNO3 to sample in beaker; let sit for a few minutes in order for sample to dissolve.

Sonicate and shake the 30 ml bottle of “Creaser Ta Gel” for at least 5 minutes.

In the cap of the beaker, mix 3.5 µl of Creaser Ta Gel to 1.5 µl of 1N H3PO4.

Load sample with pipet and clean tip onto middle of outgassed Re center filaments (loader will take up to six filaments); take current up to 1A and allow to dry flat on the filament.

Load Ta gel – H3PO4 mixture onto sample spot and allow to dry flat on the filament. Once sample is evaporated, take filament current up at a rate of ~0.2A per second to just over 2.5A, or when filament just starts to glow. Immediately turn off current.

#### Sm (Birck Ta slurry):

Outgas Re center filaments at 4A for 60 minutes.

Sonicate and shake the 30 ml bottle of “Birck” Ta slurry emitter solution for at least 5 minutes.

With the 0.5-10 µl pipet and a clean tip, add 4 µl 1M HNO3 to sample in beaker; let sit for a few minutes in order for sample to dissolve.

Load 4 µl of “Birck” Ta slurry emitter solution with pipet and clean tip onto middle of outgassed Re center filament and allow to air dry flat.

Load 2 µl of sample (that is half the solution) with pipet and clean tip on top of the dried Ta bed and allow to air dry flat on the filament. Once sample is evaporated, take filament current up at a rate of ~0.2A per second to just over 2.5A, or when filament just starts to glow. Immediately turn off current.

**Standard values**

#### U500

235U/238U = 0.999698 238U/235U = 1.000302 234U/236U = 6.862252

234U/238U = 0.010422 234U/235U = 0.010425 236U/234U = 0.145724

236U/238U = 0.001519 236U/235U = 0.001519

#### U010

235U/238U = 0.010140 238U/235U = 98.61932 234U/236U = 0.794420

234U/238U = 0.0000546 234U/235U = 0.005390 236U/234U = 1.258779

236U/238U = 0.0000687 236U/235U = 0.006785

#### SRM981 (NBS)

206Pb/204Pb = 16.937096 207Pb/206Pb = 0.914640 208Pb/207Pb = 2.370441

207Pb/204Pb = 15.491345 208Pb/206Pb = 2.168100 206Pb/207Pb = 1.093326

208Pb/204Pb = 36.721317 204Pb/206Pb = 0.059042 204Pb/207Pb = 0.064552

#### SRM981 (Todt)

206Pb/204Pb = 16.935600 207Pb/206Pb = 0.914585 208Pb/207Pb = 2.369392

207Pb/204Pb = 15.489100 208Pb/206Pb = 2.167010 206Pb/207Pb = 1.093392

208Pb/204Pb = 36.700600 204Pb/206Pb = 0.059047 204Pb/207Pb = 0.064562

#### SRM982 (NBS)

206Pb/204Pb = 36.738453 207Pb/206Pb = 0.467071 208Pb/207Pb = 2.141345

207Pb/204Pb = 17.159457 208Pb/206Pb = 1.000160 206Pb/207Pb = 2.141002

208Pb/204Pb = 36.744318 204Pb/206Pb = 0.027219 204Pb/207Pb = 0.058277

#### SRM983 (NBS)

206Pb/204Pb = 2695.4178 207Pb/206Pb = 0.071201 208Pb/207Pb = 0.191275

207Pb/204Pb = 191.92453 208Pb/206Pb = 0.016190 206Pb/207Pb = 14.04475

208Pb/204Pb = 36.708895 204Pb/206Pb = 0.000371 204Pb/207Pb = 0.005210

#### La Jolla (UCSD) JNdi-1 (Japan) Ames (MIT)

142Nd/144Nd = 1.141826 142Nd/144Nd = 1.14186 142Nd/144Nd = 1.141818

143Nd/144Nd = 0.511858 143Nd/144Nd = 0.512102 143Nd/144Nd = 0.512096

145Nd/144Nd = 0.348319 145Nd/144Nd = 0.348410 145Nd/144Nd = 0.348410

146Nd/144Nd = 0.7219 146Nd/144Nd = 0.7219 146Nd/144Nd = 0.7219

148Nd/144Nd = 0.241572 148Nd/144Nd = 0.241572 148Nd/144Nd =

150Nd/144Nd = 0.236428 150Nd/144Nd = 0.236403 150Nd/144Nd = 0.236418

#### SRM987 (NBS) Ames Sm-1b (DTM)

88Sr/86Sr = 8.375209 152Sm/147Sm = 1.783078

87Sr/86Sr = 0.710248 149Sm/147Sm = 0.921600

84Sr/86Sr = 0.056490 148Sm/152Sm = 0.420470

86Sr/88Sr = 0.1194 149Sm/152Sm = 0.516887

150Sm/152Sm = 0.276016

154Sm/152Sm = 0.850569

## Mass Spectrometry

#### Pb common (Re center with Si gel):

Warm-up center filament 2.1A in 10 minutes; for common Pb 3V beam by 2.2-2.3A.

Load “Pb Static Ax-H4” motor positions

Align Ax, H2, H3, H4 with 204Pb in Axial; align H1 on H2 with 205Pb in Axial

Run around 2.3A center

Method is “Pb Static 200 ratio”

#### Pb radiogenic (Re center with Si gel):

Warm-up center filament 2.1A in 10 minutes

Load “Pb+UO2 Faraday H4-L2” motor positions

Align H2, H3, H4 with 204Pb in Daly; align H1 on H2 with 205Pb in Daly

Run around 2.3A center

#### UO2 (Re center with Si gel):

Warm-up center filament 2.5A in 30 minutes.

Same alignment as Pb radiogenic

Run around 2.8A center

#### U (Re center with graphite):

Warm-up center filament 5A in 15 minutes.

Load “U234-235-238 Daly-H3” motor positions

Align 235U in H1, 238U in H4 with 234U in Axial

Method is “Daly U234-235-238”

#### Rb (Re center with Ta2O5):

Warm-up center filament 1.5A in 15 minutes.

Same alignment as Sr

Run around 1.5-1.7A center

Method is “Rb Static 86Ax”

#### Sr (Re center with Ta2O5):

Warm-up center filament 2.8A in 30 minutes; 3-4 V beam by 3.0-3.2A.

Load “Sr Dyn S1 L2-H3” motor positions

Align L3, Ax, H1, H2 with 86Sr in Axial; align L2 to Ax with 87Sr in Axial; align H3 to H1 with 85Sr in Axial

Run around 3.3A center

Method is “Sr Dyn no H3” or “Sr Dyn beam interp”

#### Sm (Re center):

Warm-up center filament 3.4A in 15 minutes.

Load “Sm Static 148Ax” motor positions

Using Ce-Pr-Nd-Sm-Gd collector alignment solution, align L3-H6 with 148Sm in Axial

Run around 3.4-3.5A center, between 1650-1750C; get 200mV of 147 and let it grow in with noV method

Method is “Sm Static 148Ax CeO 100 ratio”

#### Nd (split on Re sides):

Warm-up center filament 5A, side filaments 2A in 30 minutes.

Run around 2.4A side

Method “Nd Dyn 3 Seq RG”

Load “Nd Dyn 3 Seq” motor positions

Using Ce-Pr-Nd-Sm-Gd collector alignment solution, align L2 thru H5 with 143Nd in Axial; align L3 with 142Nd in Axial; align H6 with 144Nd in Axial

Method “Nd Dyn 4 Seq RG”

Load “Nd Dyn 4 Seq” motor positions

Scanning 142.5 to 144.5; split difference in alignment symmetrically between two peaks.