$^{229/230}$Thorium recovery procedure from 2 L samples on iCAP

Updated 5/30/2019

Supply List (see suppliers & cat. No at end):

- 50 mL cleaned beakers
- Watch glasses (same # as beakers)
- Forceps (plastic so will not corrode with strong acid)
- Sharpies/marking pens
- 3 ml syringe (no rubber plunger) – 1 per sample
- 0.2 micron PES filter – 1 per sample
- 5 ml pipette and tip (tip reused so not needed for every sample)
- 2 mL ICP vials & lids – 1 per sample
- 20 ml scintillation vials – 1 per sample
- lab notebook & pen

Chemicals:

- 8 M HNO_3/10% H_2O_2 solution – 10 ml per sample
  - 1L (covers ~100 samples)
  - 500 ml ACS Plus grade Fisher concentrated Nitric Acid
  - 100 ml 30% Reagent grade Fisher Hydrogen Peroxide
  - 400 ml Milli-Q water
- Clean-room or ICP lab pure water
- ^{229}Th spike solution in 5% nitric – 1 ml per sample
- ^{229}Th/^{230}Th running solution (described later)
- 20% (~2M) HCl cleaning bath

Chemical Volume needed: Nitric = (# of samples) x 5 ml
Peroxide = (# of samples) x 1 ml

Preparation:

Pre-wash all 50 mL beakers & watch glasses

Option 1 (3 overnights): Q rinse > micro bath overnight > Q rinse > 2 M (20%) HCl bath overnight > Q rinse > dry

Option 2 (1 overnight): Rinse (sink fine) > dishwasher with dishsoap > Q or RO rinse > 2M (20%) HCL bath overnight > Q rinse > dry

Jessica 4/4/2019 12:39 PM
Comment [1]: Tested with 1M solution and full digestion was not achieved and messed up results

Jessica 4/4/2019 12:42 PM
Comment [2]: Should not be made up too far in advance as peroxide degrades over time – should have bubbles in it and immediately turn the filter white. If not add a drop or two of 30% H2O2 to the beakers when dissolving

Sample Preparation

1. Label 50 mL beakers with sharpie in 2 places.
2. Dismount 25mm filter & place in labeled beaker (covered). Make notes of any that are broken, missing pieces, strange colors, etc.
3. Dissolve MnO_2 precipitate with 10 mL of 8M HNO_3/10% H_2O_2 solution pipetted into the beaker and cover with a watch glass. If needed rinse the mylar with the first 5 ml portion to get all precipitate. Discard the mylar & foil.
4. Once a tray is complete move to weighing room. TARE the beaker with watch glass on the balance. Add 1 mL of ^{229}Th/sample (~70 dpm/g) – record weight.
5. Place covered samples (entire tray) in sonicator and sonicate samples for 20 minutes. Allow to stand covered overnight.
6. Label both 2 ml ICP vials and 20 ml scintillation vials with sample names
7. Next day use a syringe to take up solution. Attach 0.2 micron PES filter and transfer approximately 0.5 mL of sample into the ICP vial (filtered to minimize potential clogging of capillary tubing during sample uptake by iCAP). Do not reuse syringes or filters – discard after use.
8. Add 1.5 mL of Q-clean water to labeled 2 mL vials for iCAP. Cap and invert to mix dense acid with water.
9. Transfer (pour) remaining unfiltered solution to 20 mL scintillation vial for storage until analysis is complete
10. Rinse used beakers before loading dishwasher, rinse watch glasses with Q or RO water and place in micro bath overnight. Discard filters if remaining so the don't clog the dishwasher.

*Fisher Plus or equivalent grade acid and reagents should be used throughout

Jessica 4/4/2019 12:45 PM
Comment [3]: If mylar has precipitate on it stick to the side of the beaker so it gets rinsed, or immediately rinse into beaker as in step 3

Jessica 4/4/2019 12:22 PM
Comment [4]: Can use a plastic bag just laid over

Jessica 4/4/2019 12:42 PM
Comment [5]: Takes ½ hr or less to do a tray of 24 samples

Jessica 4/4/2019 12:29 PM
Comment [6]: Because are looking at the ratio the exact amount does not matter. 0.5 ml seems to give a good number of counts once diluted on the iCAP.

Spike/Running Standard Preparation

1. Weigh out ~10 g of 5% HNO_3 (Ultrex)
2. Weigh out ~ 5 g of ^{229}Th solution (same as you've used to spike your samples)
3. Weigh out ^{230}Th as follows depending on its activity:

^{230}Th Activity (dpm/g)	Weigh out (g):
~ 10	~ 10
~ 50	~ 5
~ 100	~ 1

4. Use this solution to tune the instrument and measure this solution periodically throughout the iCAP run

Supplies and Suppliers

Chemicals:

Name	Grade	Supplier	Cat. No.	Notes
Nitric Acid (concentrated)	ACS Plus	Fisher	A200S121	4x2L, also comes in smaller volumes
Hydrogen Peroxide	30% Stabilized	Fisher	H323500	500 ml – if using immediately could probably get cheaper non-stabilized, but this is what we use

Supplies:

Item	Supplier	Cat. No.	Notes
3 ml Plastic syringe	Fisher	03-377-21	3 ml - 100/pk, no rubber plunger, luer-slip (DO NOT GET LUER LOCK, they won't work with filters)
PES Syringe filters, 0.22 micron, 13 mm	Sterlitech	PES0213500	Come in packs 50-1000. Smaller size, in a large bag (not sterile) – LEAD TIME usually a few weeks to month!!!!
(alternate) PES syringe filters 0.22 micron, 25 mm	Fisher	9928064	GE Healthcare Whatman Uniflo Syringe Filter. Slightly cheaper but come individually wrapped, annoying to open and more waste. A good alternative if lead time for Sterlitech is too long.
20 ml Scintillation vials	Fisher	03 341 72C	DWK Life Sciences Wheaton scintillation vials, HDPE, no cap liner, case 500.
5 ml Filtered pipette tips	Fisher	21 377 512	5 ml Finntip filtered pipette tips – filter important to protect pipette from strong acid
ICP vials – 2 ml	Fisher	02 681 343	2 ml Freestanding non-sterile microcentrifuge tubes
ICP vial screw caps	Fisher	03 313 83A	Prices vary by top color, A is clear – color part is not on inside. No o-ring.