Pb-210 analysis instructions

Updated 04/13/22 by EL, 3/30/2018by JR, 1/6/2015 by EFE; 5/23/2007 by TMD

The "lead lab," OTB 302, falls under special UW safety regulations because it is a certified radioactive chemistry lab.

- Chuck is the PI for this lab
- Andrea Ogston is the Lab Radiation Safety Agent; her cell (406-250-3405) is posted outside the door. Call if you have problems.
- Evan Lahr is the Chemical Hygiene Officer for the Lab, his cell is (612-719-8048) and is posted outside the door.

- No work may be done in the lab without the presence of someone who has passed the UW Radiation Safety training course.

- Personal protective equipment must be worn at all times. This includes closed-toe shoes, a lab coat, safety glasses or prescription glasses, and appropriate gloves (latex/nitrile for general work and rubber for acid baths).

- Record your lead run in the Radiation Safety Survey binder at the end of the day. If you are unsure of the radioactivity amounts, leave that space blank (but be sure to include your name, date, and number of samples).

- No food or drink is allowed in the lab at any time.
- In general, tools and supplies that are in the lead lab should remain in the lead lab
- After your lead run, notify a member of the Sed Lab if any supplies need re-stocking. We are happy to order more, and this will ensure a successful run for the next user.

General schedule for a set of 24 samples:
Day 1 (1-2 hrs): prep and dry samples
Day 2 (1-2 hrs): crush and weigh samples; make planchets
Day 3 (5-6 hrs): wet chemistry work
Day 4 (~2 hrs): finish wet chemistry work and dishes; start alpha counting
Day 5 (1-2 hrs): finish alpha counting for first half of samples; start second half
Day 6 (~1 hr): finish alpha counting for second half of samples

Scheduling considerations:

- Day 3 wet chemistry work must be completed before leaving the lab for the day; there is an opportunity for a lunch break partway through the process.
- Once the planchets have been immersed toward the end of Day 3 work, they must be removed 20-24 hrs later
- Alpha counting runs for ~24 hrs

- Sample prep and alpha counting can be done several days/weeks before/after the wet chemistry

Sample preparation:

1) Need **5 g of dry sediment from each sample** for analysis.
   Suggestion: Weigh out ~20 g of wet sediment.
   Need the wet/dry weight of sediment for porosity calculation.

   Specifically: **Label** the bottom of an aluminum pan with core information.
   Weigh the aluminum pan; record weight.
   Add wet sediment to pan; record total weight (for pan & wet sediment).
   Dry this sample in an oven (~60° C) for at least 24 hours.
   Remove sample from oven and allow to **cool** (can use desiccator).
   Weigh the cool pan with sediment and record weight.
   Crush sediment using a mortar and pestle until there are no clumps larger than a pea.
   Weigh 5 g of sediment (±0.05 g) into a HNO_3 washed 150 mL beaker (these beakers live in the lead lab)
   → *Each beaker must be labeled with a number (e.g., 1-24)*
   → *Write down the beaker number, sample info, and weight in your lab book*
   Store crushed sediment in small, labeled sample bag.

2) Need **silver planchets** (1 per sample) coated with *red insulating varnish* on one side.
   Soak the planchets in a small beaker of acetone to remove residues.
   (Leftover acetone should be left in the hood, with the sash down, for evaporation)
   Remove the planchets with tongs, rinse with DI water, and allow to dry.
   Arrange the planchets on printer paper in the sed lab fume hood.
   Spray with **2** light coats of varnish; allow to dry **5** minutes between coats.
   Handle the planchets with gloves for the remainder of the process.
   Punch holes in planchets with the tiny punch in the sed lab.
   Store your planchets in a labeled, covered petri dish.

3) Need acid-washed 150mL beakers (HNO_3 bath), plating jars, and centrifuge tubes (HCl bath).
   Ensure there are enough glassware and centrifuge tubes (one of each per sample) washed **BEFORE** starting your run.

4) Make sure that all your acid and spike containers are full. (They should be full from the previous run.)
   Note the date, activity (dpm/mL) of the spike solution you are using in your lab book.
   There should be a brown glass container with pump of 15.8 N HNO^3.
   There should be a brown glass container w/ pump of **<u>6 N HCl.</u>**
   Note: This is a dilution of the 12 N HCl (500mL H_2O and 500 mL HCl).
   There should be 2 plastic Nalgene squeeze bottles with **<u>0.3 N HCl</u>**.

Note: Dilution directions are on bottle. Fill to black line and add 50 mL 6N HCl.

Lab Procedures: ***always wear lab coat, gloves, and safety glasses***

Suggestion: Hotplates can be turned on immediately to begin warming up.

1) Spike each beaker (HNO_3 washed 150 mL beaker containing 5 g +/- 0.05 g of dried, crushed sediment) with **1 mL of ^{209}Po spike** using **calibrated Eppendorf pipette**.
   Use each pipette tip no more than **5** times.
   Used pipette tips should be disposed of in the Dry Radioactive Waste Box.

2) Put away the spike and Eppendorf pipette.

3) Add a clean, acid-washed, glass stirring rod to each beaker.
   Each stirring rod will stay with a specific sample for the entire run.

4) Add ~10 mL of 15.8 M HNO_3 to sample (one pump from the brown HNO_3 bottle)
   Stir and break up any sediment chunks with the stirring rod

5) Rinse down walls of beaker and glass stirring rod with small amount of DI H_2O.
   (Use just enough to clean the walls and stirring rod; using too much will increase cooking time)

6) Place beaker on hot plate under the fume hood.
   Note: Set beakers to about 3.5 on the dial.
   Any more than a 4 setting will make the fluid boil and the sediment splatter.
   If samples DO splatter, *turn down the heat*

7) Put away the HNO_3 and the DI water (the rest of the process uses only HCl)
   Take out the 6 N HCl and 0.3 N HCl.

8) Bring sample to dryness keeping in mind:
   No splattering / cook until moisture is gone / don't overcook
   > When dry, no fumes should be seen leaving the beaker
   > Once dry, take off the burner; ^{209}Po volatilizes easily and can be driven off from the sample

9) While beaker is still warm, add 10 mL of 6 N HCl; use stirring rod to break up clumps.
   Rinse walls of beaker and rod with 0.3N HCl (again, use just enough)

10) Place sample back on hot plate and bring near dryness keeping in mind step 8.

11) Pull out HCl-washed centrifuge tubes (most are pre-labeled) for each sample number.

(Corning tubes with black text fit orange lids; Falcon tubes with blue text fit blue lids. It's best to use one kind of tube/lid to keep the centrifuge balanced.)
Label plating jars (sharpie) and set on cabinet top in from of stirrers.
Place an HCl-washed magnetic stir bar in each.
Prepare silver planchets (*this can be done at any time during the run*).
> Punch hole in planchet (near the top is best)
> With a Fisher Scientific black pen, write sample information on varnished side.
> Cut ~ 10" of fishing line for each planchet and thread it through
suggestion: wrap several loops of line around two nails spaced at desired length on board; cut bundle at each nail

12) Once sample is dry again (this is the second time), add a small amount (<5 mL) of 6 N HCl and let sit for a few minutes. Stir sample to break up any clumps.

13) Rinse sample from the beaker into a centrifuge tube using 0.3 N HCl.
Fill tube to about 35-40 mL.
Tubes go into the centrifuge in batches of 8. Each batch of 8 samples must be filled to the same level.
Put 5 mL of 6 N HCl into the empty beaker to soak. Keep rod with beaker.
Place the beaker with 15 mL added 0.3 N HCl on hot plate turned down to LOW.

14) Centrifuge the samples in batches of 8 (don't forget the lid) for 10 minutes at 3000 rpm (3.0 on centrifuge setting).
Make sure the tubes have equal amounts of fluid, as an off-balance centrifuge is BAD.
If you don't have 8 samples, fill extra tubes with DI water to the same level.
If centrifuge is vibrating excessively at 3000 rpm, decrease to 2000, then 1000, then off and notify one of the lab supervisors.

15) Pour supernatant from tubes into their respective plating jars.

16) Repeat steps 13-15 **twice** more; scrape all material from beaker walls with glass rod.
On the last of 3 soaks, use 0.3 N HCl, not 6 N.

17) Once three centrifuges are completed for each sample, about 105-120 mL of yellow supernatant should be in the plating jar. Place plating jars on the magnetic stir plates set at LOW. Add L-ascorbic acid until the yellow color is gone.
Note: L-Ascorbic acid can be added to the empty jar in step 11.
If organic content is high, yellow color will not disappear. Do not keep adding L-Ascorbic acid to the jar.

18) Suspend labeled silver planchet into designated plating jar with fishing line.
Keep in mind the following:
Planchet should be fully submerged.
Planchet should not be in contact with the side of the jar.
Stir bar should not strike the plate at all.
Lid should be on tight or fishing line may slacken.

Note in lab manual the date and time that you put the silver planchets in jars (and when you remove them).
Planchets should remain in the supernatant no longer than 24 hr. (20-24 hr)

19) Complete post-lab procedures before leaving for the day.
20) Remove planchets 20-24 hours later. Rinse with DI water and let dry for counting. Finish doing dishes (see post-lab procedures).

Post-Lab Procedures:

1) Refill all acid bottles and the spike to full levels, regardless of how you found them.

2) DISHES-You must do all your dishes.
Remove any dishes that are currently in the acid baths.
Rinse them with DI water and let dry in the sink rack. Then put away!
Wash beakers and stirring rods immediately after run and place in the HNO_3 acid bath.
Wash centrifuge tubes and lids (sediment goes down the sink with the radioactivity label) and place both tubes and lids in the HCl bath.

3) Any empty acid containers should have the labels blacked out.
Fill with H^20 and place in the acid bath fume hood to vent overnight.
If you see bottles like this in the hood, dump out the water and place in the designated cardboard box for disposal.

4) Remember to sign the Radiation survey book and record the sink disposal and dry waste for your run. (This can be done when you remove samples)

5) Once planchets are removed, plating jars should be washed and placed in the HCl acid bath with magnetic stir bars. The lids to the plating jars should be rinsed (NOT SOAKED) and should NOT go in the acid bath.

Counting Procedures:

1) Turn on detectors
> **top** – power switch back left
> **bottom** – power switch front right

2) Turn on vacuum (on floor beneath detectors)
> power switch back left

3) On desktop, open Maestro

4) On top panel choose bin from drop down menu

5) Acquire → Clear

6) Service → Sample Description
Ex. BRG103_000-002

7) Place disc in detector bin, silver side up
> **remember to use tongs and not touch with hands**

8) Turn pumps on detector
> Ensemble; Acquire → MCB Properties → Alpha → Vacuum State-Pump
> Plus**;** turn pump knob

Once vacuum status < 100 mT:
Plus bin 2 won't give correct vacuum, wait until 1 & 3 are ready

9) Acquire → MCB Properties → High Voltage – On
HV/Pulsar red light will go on

10) Run count, click "Go"

11) Count for at least 80,000 counts (~22-24 hours)
No upper limit

12) When counts are done, click "Stop"

13) Save file as;
Cruise_Core_UpperDepth-LowerDepth
e.g. AMAZ3_TP-20_0-2
The file name must contain exactly two underscores as shown, and a hyphen separating the depth intervals