Sediment Sample Prep for Carbon and Nitrogen Analyses at UC Davis

IMPORTANT SAFETY INFORMATION

You must wear **safety glasses** and **gloves** when working in the lab. All work with solvents must be done in a **fume hood**. As with all chemistry labs, you need to wear appropriate clothing, including close-toed shoes, long pants, long sleeves (or a lab coat), etc.

Don't forget to write down all your steps (and/or mistakes) in the lab notebook!

If any of the methods are unclear, please check the ^{13}C and ^{15}N website page for UC Davis Stable Isotope Facility (http://stableisotopefacility.ucdavis.edu/13cand15n.html).

You can also read the literature in Limnology and Oceanography: Komada et al. 2008 Carbonate removal from coastal sediments for the determination of organic carbon and its isotopic signatures, $\partial^{13}C$ and $\Delta^{14}C$: comparison of fumigation and direct acidification by hydrochloric acid.

If you have further questions, Erin Ellis does the same analysis in the Ingalls laboratory (ellise@u.washington.edu).

1. Prepare your sediment samples by first drying and grinding them to fine particles (See laboratory methods "Sediment Sample Desalting and Drying").
2. Prepare for "vapor-phase acidification" by gathering these items:
    a. Ag (silver) capsules + capsule tray
        i. Note: do not use tin capsules as you will later have issues of corrosion, they must also have been combusted (but not combusted more than once otherwise they become brittle)
        ii. Capsule tray is most likely located in an acid bath so it needs to be removed, rinsed and placed into a fume hood covered with Kim-wipes (to eliminate dust) and allowed to dry overnight
    b. Scoopula
    c. DCM or methanol squirt bottle
    d. Kim-wipes
    e. Lab notebook and pen for documenting your masses
    f. Tweezers
    g. Metal capsule stabilizing tray (most likely already in the "scale room")
    h. Teflon plate
3. Move all of these items into the "scale room"
4. Clean the surfaces of the table with DCM to eliminate that as a potential contamination source
5. Clean the measuring plate located in the scale, the tweezers, scoopula and stabilizing plate with DCM as well
6. Use the tweezers to grab one of the Ag capsules and place it on the scale to obtain the mass
7. Tare the scale
8. Remove the capsule and place it onto the stabilizing plate

9. Using the scoopula, carefully scoop out the appropriate amount of sample (depending on sample type) and place it into the capsule
    a. Note: due to static issues, this can potentially be very difficult and fatal to your sample if you are not careful.
10. Use the tweezers again to place the capsule onto the scale to obtain the mass of your sample
11. Remove the sample-filled capsule from the scale and place it onto the Teflon plate in the appropriate spot (correlating to your sample list in your lab notebook, which will be important when transferring samples to the 96-well tray)
12. Clean the scoopula, tweezers and stabilizing tray with DCM
13. Repeat steps 6-12 for each of your samples
14. Return to your fume hood
15. Using a short glass Pasteur pipette, transfer a single drop of Milli-Q water to each sample-filled capsule
16. Then place the Teflon plate into the desiccator and close the lid
    a. The small beaker at the bottom of the desiccator needs to be filled with 25 mL of 10M HCl
17. Record your starting time
18. Choose the duration of your "vapor-phase acidification" based upon the time of samples being prepared (Recommended amount is 8 hours, and no more than 24 hours)
19. Record your ending time and remove samples from the dessicator
20. Place samples in a drying oven at ~60° C at least 24 hours
21. Remove samples and record how long they were drying for
22. Repeat steps 6-12 for all samples as well as blanks and standards to be included in the analyses (so you have acid-fumed and non-acid fumed copies of each sample)
23. CAREFULLY pinch closed all silver capsules so they resemble a ball. Fold capsules with tweezers and take care not to spill any of the samples. Folding in corners first may help with this process. If the capsule is too brittle or too full it may split. If you feel that your sample might be compromised during shipping, place into another silver (or tin if no sliver is available, just note in lab notebook) capsule. If you are unsure of the correct size or shape, check UC Davis's sample preparation website.
24. Place each sample in an individual spot in a 96-well tray. Each spot is labeled with a letter and number which UC Davis needs filled out for each sample. Record the number and letter of the sample as you place them in the tray.
25. Fill out all appropriate paperwork found on UC Davis's web page.
26. Enclose a paper copy of all the paperwork in your box with your 96-well tray.
27. Email UC Davis staff the day you mail out your samples that you have mailed your samples and include an electronic copy of the paperwork.