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isotopeprepo.pdf | A. Myrbo

Organic matter isotope preparation

Lake sediment samples are prepared for analysis of stable isotopes of carbon (and nitrogen; see below) in organic matter by treatment with an acid to destroy carbonate minerals that may interfere with the mass spectrometer. The acid is rinsed out of the sample by repeated centrifuging and decanting until no further vinegary smell is detected and pH is neutral.

Acidification can solubilize labile carbon compounds (which typically are enriched in $\delta^{13}C$; M. Talbot, pers. comm.) and thus change the isotopic value of the bulk carbon. Minimal treatment is thus recommended, unless very resistant carbonates (e.g., dolomite) are present. One hour at room temperature is frequently sufficient to remove all carbonate from the sample (some procedures recommend as much as 24h at 50°C).

Acidification can also convert one N species to another, which can result in fractionation and/or loss of N. See Teranes and Meyers 2003, who note a change in $\delta^{15}N$ of up to 8‰ due to acidification. The authors recommend that nitrogen isotope samples not be acidified. Acidification is still a common practice because of the convenience of running a single sample for $\delta^{13}C$ and $\delta^{15}N$ on a mass spec.

Organic matter stable isotopic analysis is not currently available at the Minnesota Isotope Lab.

Procedure:

1. Put wet or dried sediment into 50mL centrifuge tubes. The mass spec takes very little carbon to produce a number, so (depending on organic carbon content) a 50-100 milligrams may be sufficient.
2. Fill to the 50mL mark with acid solution. 1N HCl is the typical dilution.
3. Leave samples loosely capped 1 hour (some researchers choose different times, up to 24 hours; the emphasis should be on uniform treatment of all samples from a given core), shaking or vortexing once or twice during that time if possible.
4. Spin samples down in the centrifuge. 5 minutes at 3000 RPM should be sufficient, but settling should be gauged by eye: the supernatant (overlying liquid) should be clear, not cloudy. The supernatant may be colored (e.g., by the solution of organic compounds), but this is easy to distinguish from cloudiness caused by clay particles remaining in suspension. If the supernatant is cloudy, centrifuge longer. If it is clear, move on to the next step.
5. Decant either by pouring off the supernatant or “sipping” it using a vacuum-and-flask setup (see grain size SOP). In either case, be careful to stop pouring or sipping liquid before fine particles are swept up and lost.
6. Vortex before adding rinse water. The mixer works better when there is little overlying liquid. Occasionally it is necessary to break up the sediment pellet with a spatula; do this gently to avoid breaking sediment components.
7. Add deionized water to the 50mL mark. Some researchers use regular house DI water, but some prefer to use the high-purity Milli-Q DI.

8. Centrifuge again, decant, rinse. After the third rinse, waft the air overlying the open centrifuge tube to your nose. (The acid will be dilute enough at this point to not be harmful.) If you can detect the vinegary smell of the acid, continue to rinse. Repeat this sequence until the acid smell is no longer present. Alternately, you can use pH paper to determine when the solution has become neutralized. Chloride can interfere with the mass spectrometer, so be sure it has all been rinsed out of the sample before stopping.
9. Decant last rinse water but do not refill. Freeze and freeze-dry sediment. Tip sediment into a mortar and grind to homogenize.

Materials:
50mL centrifuge tubes (multiples of 8)
Hydrochloric acid solution
Stainless steel spatula
Acid waste bucket
Soda ash for neutralizing acid

Equipment:
Brinkmann 3810 benchtop centrifuge (holds sixteen 50mL tubes)
Vortex Jr. mixer
Water bath if temperature above ambient is desired

Safety:
Lab coat
Gloves
Goggles
Fume hood