

Standard Operating Procedures for Grain Size Analysis Sample Preparation Developed and written by Laura Triplett, October 2002

This SOP was developed on and for Lake St. Croix sediment samples to be analyzed by the IRM laser diffractometer. Procedure may vary for different sample types and analysis procedures.

Organic matter digestion

Preheat water bath¹, prepare HNO₃ solution.

Weigh ~3 grams of wet sediment into 600 mL beaker.²

Place beakers in 85°C water bath and add ~20mL of H₂O₂ (30% concentration) to each. This reaction can be violent and sporadic, so monitor the beakers and disperse excessive bubbles with a spray of DI or methanol. Add more H₂O₂ as needed to keep the reaction active.

When the H₂O₂ has stopped reacting, probably by 30 minutes, add ~2mL of HNO₃ (1:1 concentration of acid to DI, or about 11M HNO₃) to each beaker. Let that sit for 10 minutes.

Remove beakers from water bath and rinse sample into centrifuge tubes with DI and methanol.

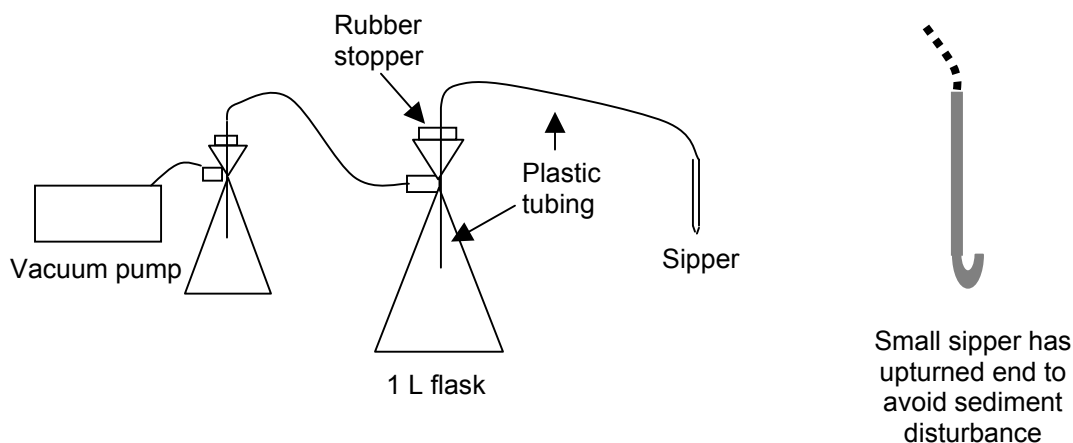
Centrifuge – 3 steps

- i. Centrifuge at 3500 rpm for 35 minutes.³ Decant, rinse and mix (see explanation below). Put supernatant in acid waste.
- ii. Centrifuge at 3500 rpm for 35 minutes. Decant, rinse and mix. Put supernatant in acid waste.
- iii. Centrifuge at 3500 rpm for 35 minutes. Decant. Put supernatant down drain. Move immediately to biogenic silica digestion, or place in refrigerator if continuing digestions later. Avoid more than 24 hours delay between the digestions.

Decant, rinse and mix (applies to all steps)

Do not pour supernatant out of centrifuge tubes, but instead use sipper mechanism hooked up to the vacuum pump (see diagram). First use the larger glass tube to draw down most of liquid in each beaker. Then use the smaller plastic sipper (from titration kit, modified) to remove the final milliliters from just above the sediment. Take care to preserve all the sediment in the centrifuge tubes. Mix well, and fill the tubes with DI water. Mixing may be accomplished by using the vortex touch mixer and/or by shaking by hand. The touch mixer is most effective if you add only a few milliliters of liquid, cap and mix, and *then* fill tube to the top.

Sipper setup:



Biogenic silica (BioSi) digestion

Preheat water bath, prepare NaOH solution.

Add ~40 mL of 1M NaOH to centrifuge tubes containing the samples from above steps. Mix well (as described in Decant, Rinse, Mix section above).

Unscrew caps (leaving them on the tubes) and place tubes (in a rack) in 85°C water bath for 30 minutes. Cap and shake once at 15 minutes, then return to water bath for remaining time.

Centrifuge – 3 steps

- i. Centrifuge at 3500 rpm for 40 minutes. Decant, put supernatant in acid waste (or separate basic waste).
- ii. After the first centrifuging and decanting of supernatant, add 8.5-9 mL of 0.5 N HCl (from general dispenser) to each tube using a pipetter. This will neutralize the solution and reflocculate the sediment to aid in the centrifuging process. Mix, then fill tube with DI as described above.
- iii. Centrifuge at 3500 rpm for 60 minutes. Decant, rinse and mix. Put supernatant in acid waste.
- iv. Centrifuge at 3500 rpm for 35 minutes. Decant. Put supernatant down drain with excess water.

Refrigerate or freeze after last step, depending on length of time before laser analysis.

Dispersant

The day before laser analysis, the sediment must be dispersed in solution.

Fill centrifuge tubes with sodium hexametaphosphate solution mixed at 5 g/L. Put on wrist-action shaker overnight before analysis.

Laser analysis

Pour sample into labeled beaker, rinse centrifuge tube with DI as necessary. Fill beaker to top of white label with sodium hexametaphosphate solution mixed at 0.5 g/L (different from dispersant above). If desired, pour through a 63 μ m sieve, rinse with DI back into beaker. (Think carefully before doing this! I do not recommend it.) If you will be sieving, put less HMP solution into the beaker to account for extra volume from DI rinses.

Acceptable transmittance ranges should be chosen based on sediment characteristics (trial and error). A smaller range ensures more homogenous sampling, but also makes analysis more slow and prone to errors.

Footnotes

- 1 Water bath should be in a hood.
- 2 Ideally sediment was never dried or freeze-dried because that can make it harder to disperse the clay minerals in solution. The mass needed will vary depending primarily on the % moisture and % organic matter in the sample. At least 0.5g of sediment should remain after all the digestions.
- 3 Centrifuging is complete and successful when the supernatant is transparent. There may be some yellow color in the first centrifuge after the organic matter digestion, but the water will still be transparent. If the water is cloudy, centrifuge for a longer period of time (but no longer than 60 minutes).