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Muffle Furnace

Standard Operating Procedure

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Purpose

Water, organic matter, carbonate mineral, and siliciclastic+diatom content are estimated by sequentially measuring weight loss in sediment core subsamples after heating at selected temperatures.

A compositional profile can be generated rapidly and for very low

cost. This profile is sufficient to develop a general sense of core stratigraphy and often is sufficient for correlation between cores.

The results are accurate to 1-2% for organic matter and carbonate in sediment with over 10% organic matter. In clay- or diatom-rich sediment, the water of hydration is lost during the carbonate burn, resulting in errors of up to 5% for carbonate analyses (and “false positive” carbonate content in carbonate-free sediments). If high precision (0.1%) is needed, or if sediment is in short supply, colorimetric analysis is recommended.

A nonprogrammable Lab Line L-C oven is used for the 100°C drying step. Other drying ovens are also available in the lab. A drying oven rather than a furnace is used for the 100°C step because the furnaces ramp up to temperature quickly and may overshoot 100°C by an amount that could affect the analysis.

A Fisher Scientific Isotemp Programmable Muffle Furnace is used for the 550°C and 1000°C steps. This is a multiple mode instrument capable of reaching 1125°C and controlling temperatures to better than $\pm 15^\circ\text{C}$ with a $\pm 10^\circ\text{C}$ temperature uniformity. The programmable circuit will provide the necessary corrections to maintain temperatures at established set point. This furnace holds 200 samples at a time. Another furnace, the Lindberg Blue M, is available for use as a backup, but only holds 50 samples. This is a two mode instrument capable of controlling temperatures to better than $\pm 10^\circ\text{C}$. The automatic reset circuit will provide the necessary corrections to maintain temperature established set point.

Equipment

- Ceramic crucibles
- Firebricks drilled to hold crucibles
- Sampling device (spatula, syringe)
- Desiccator(s)
- Drying oven
- Muffle furnace capable of reaching 1000°C
- Balance weighing in grams to 4 decimal places

Safety

The most obvious hazard in LOI is being burned by hot samples fresh out of the furnace. Be patient. The high-temp gloves and mitts are only good to about 350°C and can be awkward to use.

The muffle furnaces each have a thermocouple (looks like a white stick with metal protruding from the end) which penetrates in through the back of the chamber. It is easily damaged, so be careful not to bump it when adding or removing samples.

Summary

Subsamples are placed in weighed crucibles and weighed. Weight loss is measured after heating the samples overnight at 100°C to remove water, at 550°C for four hours to remove organic matter, and at 1000°C for two hours to remove carbonates. After each heating step, the firebrick holding crucibles is allowed to cool completely in the oven or furnace before weighing, or placed in a desiccator if crucibles cannot be weighed immediately. Samples

must be cool so that convection currents do not affect the balance, and kept in the oven, furnace, or desiccators so that they do not absorb atmospheric water.

Samples must not be placed in non-venting desiccators when warm.

Ash left at the end of the procedure can be saved for analysis of remaining elements and oxides.

Only one heating step can be accomplished each day because the 100°C drying time and the ramp-up and cool-down times of the furnaces are all >8-10 hours. The user should thus plan five days, ideally consecutively:

Day 1 Weigh crucibles (if necessary), subsample, and weigh sample (initial or wet weight); place samples in drying oven at 100°C (allow several hours for these steps, depending on subsampling complexity).

Day 2 Turn off oven, let samples cool, weigh (100°C, dry weight, or water loss); place samples in a furnace at 550°C for 4 hrs.

Day 3 Weigh (550°C or organic matter loss); place samples in a furnace at 1000°C for 2 hrs.

Day 4 Weigh (1000°C or carbonate loss), discard or save sample residues, wash crucibles, place crucibles back in trays, place trays

of crucibles in a furnace at 1000°C for 2 hours to completely clean the crucibles.

Day 5 Make sure crucibles are cool, remove trays from drying oven, cover with foil, and place in a desiccators for reuse (1-2hr). Label these trays as clean and ready to be reused.

Procedure

- 1) Select and weigh 25 crucibles per fire brick tray. You can prepare up to 200 samples (eight trays) for analysis at a time.
 - a) Log into the Analytical database, click LOI Requests/QUEUE” and create a new record for each separate project you are working on. Fill in Expedition, lake, year and number of samples. Then go to the LOI data page.
 - b) Never touch crucibles with your hands. Skin oils will add weight and introduce error into your results. Always use a pair of tweezers when handling crucibles. Be sure to tare the scale at the start of each tray to check for drift.
 - c) The crucibles are numbered with permanent glaze. Check to see that they are in order in the brick (see below) and that you are starting with the lowest numbered crucible in your tray.
 - d) When not burning or being weighed, the crucibles and the samples they contain must be stored in a desiccator. Make sure there is enough desiccator space for the number of samples you hope to analyze. Wait until the samples are cool ($<30^{\circ}\text{C}$) before putting them in the desiccator: the decrease in pressure upon cooling of the air in the desiccator will vacuum-seal the desiccators shut, and it'll be very difficult to open.
 - e) Always remove aluminum foil before placing trays in drying

oven or furnace

- 2) Record your crucible numbers and weights in the LOI data table of the Analytical database. Click "LOI Data," button on the home screen. Add a new record for each sample collected. The layout will highlight (in red) any weights entered that are clearly incorrect (i.e., less than or equal to the empty crucible weight or greater than or equal to the weight of the preceding burn). Care must still be taken to ensure correct values are entered into the database. You should also record the Core ID, Interval_Top, Interval_Bottom, for each sample.
- 3) Place some sample (~1-5 cc) in each crucible and weigh. Weighing should be done as soon as each tray is filled, do not wait to fill multiple trays before weighing. This is your wet weight. Record the Core ID, Interval_Top, Interval_Bottom, and Weight_wet in LOI data table, if not already entered at the time of crucible weighing.
 - a) If samples will be sent in for Lead-210 dating, pull the required samples while you have them out (and sorted) and segregate them for easy sampling when following the Lead-210 procedure.
 - b) Remember: all the calculations in the database (i.e. volume, ratios, etc) will not be calculated until the LOI process is complete (e.g. all weights [empty, wet, 100, 550, 1000] have been entered).
- 4) Heat these samples at 100-105°C overnight or for at least 12 hours in the drying oven. Refer to separate SOP for drying ovens and furnaces. This will evaporate water, and the resulting weight will be your 100°C weight. Turn off the oven and let samples cool

(until $<30^{\circ}\text{C}$), before removing and weighing. If you cannot weigh the samples immediately after they cool, place in a desiccator with aluminum foil between each tray until you can weigh them.

- 5) After weighing and recording your 100°C weight, return the samples to the furnace for a 4-hour burn at 550°C . This will burn off organic matter. The following day, after samples have cooled in the furnace, samples can be weighed. If you cannot weigh the samples immediately after they cool, place in a desiccator with aluminum foil between each tray until you can weigh them. This will prevent samples from taking in air moisture and throwing off your weights. See furnace directions below.
- 6) Record your 550°C weight in the database and return the samples to the oven for a 2-hour 1000°C burn. This will burn off a combination of carbonate material and some of the water stored in the lattice of clay minerals and diatom silica. See furnace directions below.
- 7) After cooling, record this 1000°C weight as your final measurement. You may discard the sample remaining in the crucible, or save it for another analysis.
- 8) The results are automatically calculated in the database. Look at the corresponding graph to help spot any errors.
- 9) Clean the crucibles for the next user.
 - a) Two buckets are needed and should be in or near the sink in room 680A. Fill one
 - b) Bucket with warm tap water and add soap from the bottle



labeled "Lab Soap" above the sink. Fill the other bucket with low-purity deionized (DI) water from carboy above the adjacent sink.

- c) Remove any remaining residue and place the crucibles in the warm tap-waterbath.
- d) Using a brush found above the sink, scrub the crucibles until all baked on residue is gone. Some discoloration will remain.
- e) Rinse the crucibles in the DI water bath, shake dry and place in the firebrick tray following the numbering system designated in the figure on the right.
- f) Place all washed trays of crucibles in the furnace and burn at 1000°C for two hours (in the same manner as the carbonate burn).
- g) The following day, remove all trays from the furnace, cover with aluminum foil and place in the appropriate desiccators. Use the "Cleaned 1000°C " crucible icons or label the trays "Cleaned and burned at 1000°C " between each trays.