

Loss-on-Ignition

Purpose

Water content, organic matter and carbonate content are estimated by sequentially measuring weight loss in sediment core subsamples after burning at selected temperatures.

A compositional profile can be generated rapidly and for very low cost. This 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 high clay sediment, water of hydration is lost during the carbonate burn resulting in errors of up to 5% for carbonate analyses. If high precision (0.1%) is needed, or if sediment is in short supply, coulometric analysis is recommended.

The muffle furnace is the Lindberg Hevi-Duty Type B Controller. This is a 2 mode instrument capable of controlling temperatures to better than $\pm 10^{\circ}$ C. The automatic reset circuit will provide the necessary corrections to maintain temperatures at established set point.

Procedure Summary

Subsamples are placed in weighed crucibles and weighed. Weight loss is measured after heating at 100° C overnight 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 placed in a desiccator to cool completely before weighing so that the samples do not absorb atmospheric water and so that convection currents do not affect the balance.

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

About 100 samples can be processed in 2 days of concentrated work.

Equipment

Ceramic crucibles
Firebricks drilled to accept 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 and let them cool down as much as possible before handling. The high-temp gloves and mitts are only good to about 350° C and can be awkward to use.

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

Procedure Detail

1. Select and weigh 25 crucibles per fire brick tray. You can prepare up to 50 samples (two trays) for analysis at a time.
 - a. Never touch crucibles with your hands. Skin oils will add weight and introduce error to your results. Always use a pair of tweezers when handling crucibles.
 - b. Make sure you record some notes that help you identify which sample is which in your fire brick. Most bricks have had a corner knocked off or have had an “X” or some such mark carved in a side. Put a marked crucible in a corner or with reference to the X and use that as a reference point. Keep good notes! As soon as you’ve mixed things up the data are useless.
 - c. From now on, when not burning or being weighed, the crucibles and the samples they contain should be stored in a dessicator. Make sure there is enough dessicator space for the number of samples you hope to analyze. Wait until the samples are cool (<50° C) before putting them in the desiccator: the decrease in pressure upon cooling of the air in the desiccator will vacuum-seal the desiccator shut and it’ll be very difficult to open.
2. Record your crucible weights in the LOI template spreadsheet.
3. Place some sample (~1-5 cc) in each crucible and weigh. This is your *wet weight*. Record in LOI spreadsheet. *Note:* if you use the **LOI macro** (more about which later), the samples do not have to be volumetric.
4. Heat these samples at **100-105° C overnight** in either the muffle furnace or the drying oven. This will evaporate water and the resulting weight will be your *dry weight*. Let samples cool in a dessicator for at least half an hour before weighing. Warm samples will yield inconsistent weights due to the heat-generated convection currents in the balance, so be patient.
5. After weighing and recording your dry weight, return the samples to the furnace for a **4-hour burn at 550° C**. This will burn off organics and will likely stink pretty badly for the first hour or so. Remember that the oven will have to ramp up and down from the burning temp of 550°. This adds time, so budget accordingly. When the oven has cooled down to around 50° C you can remove the samples and place them in a dessicator to cool further. Use the mitts and gloves that are stored in the drawers below the oven. Do not let the samples cool outside of the oven or a desiccator: this will allow samples to take in air moisture and throw off your weights.
6. Record your post-550 burn weights in the spreadsheet 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.

7. After cooling, record this weight as your final measurement! You may throw out the sample remaining in the crucible, or save it for another analysis. Crucibles should be washed using tap water and Liquinox, and rinsed with deionized water. Let fully air dry overnight in tray and then return to storage dessicator.
8. Use the LOI macro (created and graciously provided by D.R. Engstrom) to calculate your percent component fractions, wet and dry bulk density, etc. To use the macro [*saved on the desktop as **LOI.2.xls***], the spreadsheet that contains your LOI data must be in the following format (column titles):

Depth, Wt. Crucible, Wt. Wet, Wt. 100, Wt. 500, Wt.1000.

Or

Top depth, Bottom depth, Wt. Crucible, Wt. Wet, Wt. 100, Wt. 500, Wt.1000.

These columns and the data within them are the **ONLY** cells that may be filled in on the page, or the macro will malfunction. If you have supplementary data such as core names, notes to self, etc., cut them from this spreadsheet and put them in another sheet in the same workbook.

Save the data spreadsheet and close it. Open up the LOI.2 file. (Click "enable macros" in the warning window that pops up.) Go to Tools--> Macro--> Macros. The "macro name" line should read "A1" and be highlighted. The the first line of the section below A1 should read "LOI." Click "Run."

The next box that pops up asks for the file name (as saved on the spreadsheet), some info about the coring site (nonessential), whether your depth intervals are single or you've used two columns for top and base depths (determined by which format for column titles you used above), and whether you did a CaCO₃ (1000° C) burn. Once you've filled in and selected the appropriate options, click "OK" and the macro will perform its magic. It will probably not find your file at first, but you have the option of browsing for it.

The resulting data will be placed in a bunch of columns to the right of your original data.

For a comprehensive review of best practices and comparative LOI methods, please see [Heiri](#) et al, Journal of Paleolimnology 25, p. 101-110.