

## Loss-on-Ignition

### 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, 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, coulometric 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 2 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 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 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 desiccator so that they do not absorb atmospheric water. Samples must not be placed in a non-venting desiccator when warm.

Ash left at the end of the procedure can be saved for analysis of remaining elements as 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 (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 furnace at 550°C for 4 hr (about 0.5-1 hour per tray of 25 samples).

**Day 3** Weigh (550°C or organic matter loss); place samples in furnace at 1000°C for 2 hr (about 0.5-1 hour per tray of 25 samples).

**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 furnace at 1000°C for 2 hours to completely clean the crucibles.

**Day 5** Turn off oven, allow crucibles to cool completely, remove trays from drying oven, cover with foil, and place in desiccator for reuse (1-2 hr). Label these trays as clean and ready to be reused.

### 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. The high-temp gloves and mitts are only good to about 350°C and can be awkward to use.

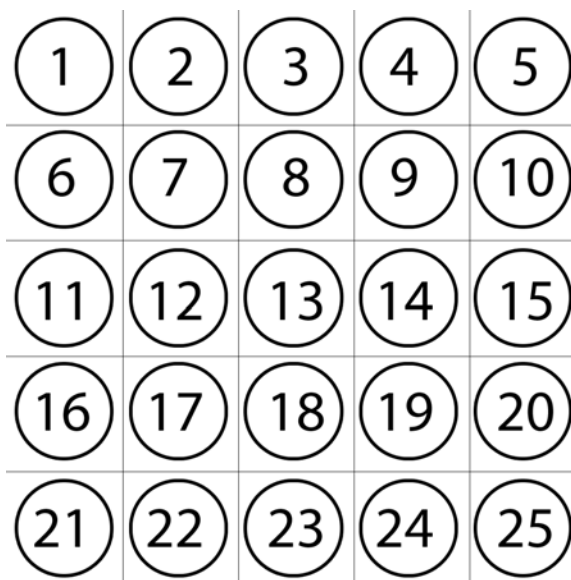
The muffle furnaces each have a thermocouple (looks like a white stick with metal protruding from the end) which penetrates in through the back of the chamber. It is easily damaged, so be careful not to bump it when adding or removing samples.

### Procedure Detail

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. 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. 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. Keep good notes! As soon as you've mixed things up the data are useless.
  - c. 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°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.
  - d. Always remove aluminum foil before placing trays in drying oven or furnace
2. Record your crucible weights in the LOI template spreadsheet [*saved in transfers/SOPs/ as LOI.Template.xls*]. This new template 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).
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 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 or for at least 12 hours in** the drying oven. This will evaporate water and the resulting weight will be your *100°C weight*. Let samples cool in the oven, until <30°C, before 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  $100^{\circ}\text{C}$  weight, return the samples to the furnace for a **4-hour burn at  $550^{\circ}\text{C}$** . 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 allow samples to take in air moisture and throw off your weights. See furnace directions below.
6. Record your post-550 burn weights in the spreadsheet and return the samples to the oven for a **2-hour  $1000^{\circ}\text{C}$  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 weight as your final measurement. You may discard the sample remaining in the crucible, or save it for another analysis.
8. Run the LOI Macro. See directions below.
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 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 water from carboy above the adjacent sink.
- b. Remove any remaining residue and place the crucibles in the warm tap-water bath.
- c. Using a brush found above the sink, scrub the crucibles until all baked on residue is gone. Some discoloration will remain.
- d. 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.
- e. Place all washed trays of crucibles in the furnace and burn at  $1000^{\circ}\text{C}$  for two hours (in the same manner as the carbonate burn).
- f. The following day, remove all trays from the furnace, cover with aluminum foil and place in appropriate desiccators. Use the “Cleaned  $1000^{\circ}\text{C}$ ” crucible icons or label the trays “Cleaned and burned at  $1000^{\circ}\text{C}$ ” between each tray so that the next person knows they are ready to be used.



### How to use the Lab Line L-C oven

1. Open the oven and place your trays on the shelving in the oven. Load in the top trays first to prevent contamination of the samples. Close the oven door
2. Turn on the oven using the switch on the front panel.
3. Set the temperature knob about  $\frac{3}{4}$  of the way between a setting of 4 and 5 to heat the oven to  $100^{\circ}\text{C}$ .
4. The oven does not have a temperature feedback control system, it is a good idea to periodically check the temperature on the oven to make sure it reaches  $100^{\circ}\text{C}$  but does not greatly exceed  $100^{\circ}\text{C}$ .

### How to use the Isotemp muffle furnace in Ramp and Soak mode

1. Open the flue on top of the furnace.
2. Turn on the Furnace using the switch on the front control panel.
3. Open the furnace and place your trays on the shelving in the furnace. Load in the top trays first to prevent contamination of the samples. 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. Close the furnace door.

4. Verify that the Run LED is not on. If it is on, press Run until the light goes out.
5. Press the following sequence of buttons in the left most column.

<u>Button</u>	<u>Top Display</u>	<u>Lower Display</u>	<u>Description</u>
Menu	No	program	Furnace is not in program mode.
UP	Yes	program	Select yes to set program parameters
Menu	1	step	The first step in the program
Menu	SP (Set Point)	styp (step type)	is a set point.
Menu	(550 or 1000)°C	sp	This is the temp from the last time the program ran.
Up/Down	(550 or 1000)°C	sp	Use the up or down keys to set to 550 or 1000°C.
Menu	20° C	rate	The ramp up rate should always be 20°C for LOI.
Menu	No	retn (return)	No return for this step,
Menu	2	step	move on to step two in the program,
Menu	Soak	styp	a soak step.
Menu	(4 or 2)	hour	This is the length from the last time the program ran
Up/Down	(4 or 2)	hour	Use the up or down keys to set to 2 or 4 hours,
Menu	0	min	0 minutes,
Menu	0	sec	0 seconds,
Menu	No	retn	No return for this step.
Menu	3	step	Move on to step three in the program,
Menu	end	styp	to end the program,
Menu	off	end	by letting the furnace cool to room temp.
Menu	yes	save	Save the program.
Menu	actual temp	set temp	You have exited the program parameters.

6. Press run twice to run the program, the run light should be solid (not flashing) and the program will automatically start to ramp up to temperature. You can see the set temperature and the actual temperature on the display.
7. Once the furnace has cooled down, you can close the flue and turn off the furnace. If the alarm LED is lit up on the control panel please notify staff, this means that the actual furnace temperature exceeded the set temperature by more than 25°C
8. Remove the bottom shelf of samples first, to prevent contamination.

### **How to use the LOI Macro**

To use the macro [*saved in transfers/SOPs/LOI as LOI.Macro.xls*], the spreadsheet that contains your LOI data must be in the following format (column titles):

Depth, Crucible Weight, Wet Weight, Weight (100°C), Weight (550°C), Weight (1000°C)

or

Depth (Top), Depth (Bottom), Crucible Weight, Wet Weight, Weight (100°C), Weight (550°C), Weight (1000°C).

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 crucible number, 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 as a **new file** (the macro overwrites the opened file) and close it. Open up the **LOI.Macro.xls** file. (Click "enable macros" in the warning window that pops up.) Go to Tools--> Macro--> Macros (or press Alt-F8). The "macro name" line should read "A1" and be highlighted. 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 CaCO<sub>3</sub> (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 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.