

LOI Procedure

This loss-on-ignition (LOI) method requires the following four measurements for every sample calculation:

Measurement 1: weight of empty 10 ml plastic scintillation vial

Measurement 2: weight of 10 ml scintillation vial with wet sample

Measurement 3: weight of 10 ml scintillation vial with dry sample

Measurement 4: weight of empty crucible

Measurement 5: weight of crucible and dry sample

Measurement 6: weight of crucible and dry sample after 550°C burn for 4 hours

Measurement 7: weight of crucible and dry sample after 1000° C burn for 2 hours

The method of calculations are very susceptible to atmospheric moisture content- negative weight losses (i.e. weight gains) can be easily generated by letting the samples sit out after drying/burning, and then weighing them. This issue can be overcome by maintaining the samples in a 105°C drying oven, and then weighing them very quickly when they are still hot. (In our drying oven it is smart to place a piece of tinfoil on the top shelf so that flakes from the top tray do not fall into the bottom and add additional weight.) This means not letting the samples sit out on the counter to cool before weighing, and weighing them as quickly as possible using tongs (you should be able to do a whole tray of 25 crucibles in 6-7 minutes).

Make sure to recalibrate the balance before you use it each time. The bubble should be leveled and the bubble should be in the center of the gray inset in the front of the balance. And then calibrated by pressing the setup button to the left of the display and then pressing enter after each prompt until it begins calibrating itself.

21	22	23	24	25
16	17	18	19	20
11	12	13	14	15
6	7	8	9	10
1	2	3	4	5

Steps:

1. Label and weigh a tray of 100 empty plastic 10 ml scintillation vials with caps on top. Label the caps as well with the numbers of the corresponding vials. This gives us **Measurement 1**.
2. Sample 1 cubic centiliter of the part of the core you want sampled. Put this sample into the empty scintillation vials.
3. Weight the plastic scintillation vile that contains each wet sample with caps on top. This gives us **Measurement 2**.
4. Remove caps from plastic scintillation vials and put them under each vial in the vial tray. Then put the tray of vials in an oven, heated at 60° C for no less than 36 hours.
5. 12 hours before you are ready to do the 550° C burn and 24 hours after you put the vials in the 60° C oven, make sure the crucibles are clean at put them in an oven heated to 105° C. (This will evaporate all of the water out of the crucibles.) You should store one tray of crucibles on the top shelf and one on the bottom. You should put the top tray in first and then the bottom tray.
6. After 36 hours at 60° C the samples should be void of all water. After at least 36 hours you should remove the tray from the heat and weigh the first 50 vials with the dry sample and the caps screwed on. This will give you **Measurement 3**. (You will return the other 50 vials to the oven at 60° C until you are ready to do the 550° C and 1000° C burns for those remaining 50.)
7. Next you will weight each empty crucible. You should label the crucibles according to lay out specified according to the above configuration. Also, the bottom tray should be labeled with a 'B' and then the number and the top tray should be labeled with a 'T' and then the number. You should remove the bottom tray first and weigh those 25 empty crucibles, then remove the top tray and weigh those 25 empty crucibles. This will give you **Measurement 4**. Before you weigh these you may want to turn the muffle furnace on and set it at 550° C. (It will take about an hour to reach that temperature).
8. Now you will put the dry sample from the scintillation vile into the corresponding crucible. You will then weight the crucible plus the dry sample. This gives you **Measurement 5**.
9. Once the muffle furnace has reached 550° C you will first put the top tray on the top shelf and then put the bottom tray on the bottom shelf. Leave these in the oven for 4 hours. After 4 hours you will take them out and place them in the 105° C drying oven. (It is helpful to immediately set the muffle furnace at 1000° C to prepare for the next burn. It usually takes a good hour to heat up to 1000° C). You should remove the bottom tray first and then the top tray. After the samples have cooled. Take the bottom tray out of the drying oven and then weigh the crucibles. This will give you **Measurement 6**. After you weight each tray of crucibles replace each tray in the drying oven. Once the muffle furnace heats to 1000° C put the top tray of crucibles on the top shelf and the bottom tray on the

- lower shelf. After you open the furnace it will probably take another 45 minutes to reheat to 1000° C.
10. After the crucibles are in the muffle furnace at 1000° C for 2 hours you should turn the furnace off, open the door and let it cool for at least a half hour. When you remove the trays you should put on a gloves as well as a face mask. It is very hot and you should be wary of other people in the area. Next, remove the trays in the order described above and place them in the 105° C drying oven until they are cool enough to weigh. You should remove and weigh the crucibles on the bottom tray first and next you may remove the top tray and weigh those crucibles. This will you give you **Measurement 7**.
 11. After you have weighed all of the crucibles you should dispose of the dried sample and clean the crucibles with water. Then either replace them in the drawer, or if you or another person in the lab plans to do another LOI, replace them in the drying oven for at least 12 hours before the next set of burns.