

Loss-on-Ignition Procedure

This procedure is intended for rock or soil samples. All materials should be homogenized, finely ground, and dried in an oven at 105-110°C for a minimum of 2 hours. Store oven-dry samples in a desiccator prior to LOI analysis.

Preheat the muffle furnace.

1. Turn on the oven. You will hear a loud beep.
2. On the diagram below the heating curve, hit the gray TEMP button. Ensure that the temperature is set to 950°C. Hit the button once more to check that the second temperature step is also set to the desired temperature. If either temperature needs to be changed, type in the desired temperature while the red light is blinking above the TEMP 1 or TEMP 2 steps. Hit enter after entering the temperature.
3. Hit START/STOP once to turn on the heating cycle. The oven will issue two loud beeps when heating begins. Ensure that the temperature starts to increase within a couple minutes.

Prepare samples for roasting

4. Wear neoprene or latex gloves.
5. Sanitized crucibles will be located in a pyrex desiccator. It is our policy to store sanitized crucibles by themselves. Locate the desiccator with empty crucibles.
6. Gently slide the lid off the desiccator, ensuring that you do not transfer any silicone grease onto your gloves. If that happens, change your gloves. Place the lid of the desiccator top-down on a counter – never place the lid greased-side down!
7. Retrieve the sanitized crucibles. Each crucible is labeled with a number on its base. Record the number and your corresponding sample number in the lab notebook.
8. First, record the weight of the crucible.
9. Then, add the appropriate sample mass to the crucible without taring the balance. Target weights for samples in each type of crucible are as follows:
 - a) Smallest (tiny) crucibles: 0.2-3.5 g
 - b) Small crucibles: 3-5 g (no less than 3 g)
 - c) Large/wide crucibles: 5-10 g (no less than 5 g)Note that the above weights are just the sample mass. You should add this to the mass of the crucible. Record the sample mass + crucible mass in a separate column in the lab notebook.
10. Repeat steps 8-9 for each sample. Every sample should have the crucible number, sample number, crucible mass, and crucible + sample mass recorded.

Roast the samples

11. Once the oven reaches the desired temperature, press HOLD.
12. Move all crucibles to the area in front of the desiccators, next the oven. Remove latex/neoprene gloves and put on safety glasses and fire-retardant gloves.
13. Using the long tongs, carefully transfer the crucibles to the muffle furnace. Close the door tightly after you are finished.
14. Once all samples are in the oven, wait until the temperature returns to 950°C. Set a timer for 105 minutes (1 hour and 45 minutes).

Retrieve the samples

15. After 105 minutes, prepare a pyrex desiccator by opening the lid and ensuring there is space for the hot crucibles.
16. Wearing safety glasses and fire-retardant gloves, use the long tongs to transfer each crucible to the pyrex desiccator. Replace the lid, but leave it cracked open for about 10 minutes to release hot air, then seal it completely.
17. Turn off the muffle furnace and ensure the door is closed.

Weigh roasted samples and calculate LOI

18. Once the crucibles are cool (~1 hour), weigh each using the Sartorius balance. Be sure to wear latex/neoprene gloves to prevent transfer of finger grease to samples. Also, be mindful to prevent transfer of silicone grease from the desiccator lid.
19. Record the crucible + roasted sample mass in the notebook.
20. Subtract the crucible mass from the crucible + dry sample mass and crucible + roasted sample mass.
21. Calculate LOI using the following equation:

$$\text{LOI \%} = [(\text{oven-dry sample wt.} - \text{roasted sample wt.}) / \text{oven-dry sample wt.}] \times 100$$