

Lake Sediment Loss on Ignition (LOI) Protocol

ALWAYS WEAR GLOVES WHEN HANDLING THE CRUCIBLES

1. Place approx. 0.5g wet sample in crucible of previously known weight
2. Weigh (W_{wet}) (for moisture content, don't bother if already have moisture content)
3. Place crucible and sample in 150°C oven overnight
4. Cool in a desiccator
5. Weigh (W_{dry})
6. Calculate moisture content: $(W_{wet} - W_{dry}) / W_{wet} * 100$
7. Cook in furnace at 550°C for 2.5 hours
8. Cool in a desiccator
9. Weigh (W_{550})
10. Calculate percent weight lost at 550°C: $LOI_{550°C} = (W_{dry} - W_{550}) / (W_{dry} - W_{cruc}) * 100$
11. Discard sediment and wash crucibles. Allow crucibles to dry overnight or heat them at 150°C in the oven for at least 2 hours before using again (that is, if you need moisture content and therefore need an accurate measurement of W_{wet} . If you already have moisture content (e.g. from freeze drying the sediment) then you can wash and towel dry the crucibles before using them again immediately).

To move samples from oven to desiccator to balance:

1. Allow oven to cool to 150-250°C (so that it's not too hot when you're removing crucibles, but no cooler than 150°C or else the samples will begin to draw water from the air)
2. USING TONGS, place crucibles in the desiccators (be very careful when removing the desiccator tops, they can stick and then fly off easily, it's easiest to open if you release the vacuum in the desiccator by turning the very top part until the tube is next to the opening and then slide the top off horizontally instead of pulling it off vertically)
3. Put tops back on desiccators, allow crucibles to cool to room temperature (this assures that they suck as little water as possible from the air when you remove them from the desiccators)
4. Once the crucibles are cool, PUT ON A PAIR OF GLOVES, carefully slide tops off of desiccators, place crucibles in egg cartons (using gloved hands) and bring to balance
5. Once you've weighed each crucible, place them back in the desiccators and put the tops back on the desiccators (unless they've already been through the 550°C step, in which case you can discard the sediment and wash the crucibles using the red brillo pad in the lab sink)

IMPORTANT: DO NOT TOUCH the crucibles directly with your fingers, as oil from your skin can change the weight.

Example lab book data sheet for loss on ignition:

Depth (cm)	Crucible #	W _{wet} (g)	W _{dry} (g)	W ₅₅₀ (g)
14	1	8.6435	8.1977	8.1871

Example Excel calculation sheet for loss on ignition, with formulas from steps 6 and 10 entered into columns 8 and 11, respectively.

1	2	3	4	5	6	7	8	9	10	11	12
Sample name	Crucible #	Crucible wt (g)	W _{wet} (g)	W _{wetsed} (g)	W _{dry} (g)	W _{drysed} (g)	Moist Cont (%)	W ₅₅₀ (g)	W _{550sed} (g)	LOI (%)	Depth (cm)
0.25-0.5	1	8.2276	8.7369	0.5093	8.2596	0.0320	93.7	8.2542	0.0266	16.9	0.25