

Loss on ignition (LOI)

1. After step 2, handle crucibles with crucible tongs only. Keep crucibles in glass bell dessicators with drierite between steps.
2. Label crucibles, if not labeled. Use high-temperature pen on underside.
3. Wash and dry crucibles in drying oven at 95°C.
4. Weigh crucibles.
5. Add 1 cm³ of wet sample (*or approximately 1 cm³ if not estimating bulk density*)
6. Weigh wet samples (*only if estimating bulk density*)
7. Place crucibles in drying oven for at least 8 hours.
8. Weigh crucibles with dry samples. Don't need to use dessicator after this step.
9. Place crucibles in furnace. Make sure crucibles do not touch each other or the furnace walls. (Use metal rack for 18 crucibles). Combust at 550°C for at least 2 hours (add more time if furnace is heating up).
10. Turn off furnace, let cool for several minutes. Place crucibles in dessicator, and let cool to room temperature.
11. Weigh crucibles with ash.
12. CONTINUE ONLY IF THERE IS EVIDENCE OF CARBONATES IN SEDIMENT (i.e., fresh sediment fizzing in HCl). Place crucibles in furnace. Combust samples at 950°C for 2 hours.
13. Turn off furnace, let cool until possible to safely remove crucibles. Place crucibles in dessicator to let cool to room temperature.
14. Weigh crucibles with ash.
15. Discard ash, wash crucibles.

INSTRUCTIONS FOR MUFFLE FURNACE:

Use metal tray (with handle) designed for holding small crucibles.

If not using metal tray, use less than two-thirds of any dimension of the chamber. Maintain a 3/4" clearance between the load and the sides of the chamber.

- If you are heating a number of small parts, spread them throughout the middle two thirds of the chamber.
- Keep objects away from thermocouple.
- Raise your load up off the furnace floor with small pieces of ceramic or a hearth plate to promote even heating.
- Use insulated tongs and mittens when loading and unloading furnace.
- Always wear safety glasses.

DRIERITE REGENERATION:

For the regeneration of [Indicating DRIERITE](#) and small lots of [Regular DRIERITE](#), the granules may be spread in layers one granule deep and heated for 1 hour at 210° C or 425° F. The regenerated material should be placed in the original glass or metal container and sealed while hot. The color of the Indicating DRIERITE may become less distinct on successive regenerations due to the migration of the indicator into the interior of the granule and sublimation of the indicator.

The temperature at which DRIERITE desiccants are regenerated is crucial in restoring DRIERITE to its original condition. Absorbed moisture is water of hydration and is chemically bound to the calcium sulfate of DRIERITE. Temperatures in the range of 400° - 450° F are required to break these bonds and release absorbed moisture. Lower temperatures, regardless of heating time, will not regenerate DRIERITE unless applied under vacuum (26" Hg, 325° F or 28" Hg, 275° F). Care should be taken not to overheat DRIERITE Desiccants. High temperatures can alter the crystal structure and render the desiccants permanently inactive.

Loss-on-ignition:

Notebook and Excel spreadsheet format:

	A	B	C	D	E	F	G	H
1	Crucible no.	Volume sediment (cm³)	Sample depth	Crucible weight	Crucible+ wet sediment	Crucible+ dry sediment	Crucible+ 550°C combusted sample	Crucible+ 950°C combusted sample
2	1	1	35	1.44	1.95	1.58	1.49	1.46

Equations for columns I, J, K, L

Bulk density = (F2–D2)/B1 = 0.14 g / cm³

% Water = (E2–F2)/(E2–D2) = 0.37/0.51 = 72.5%

% LOI = (F2-G2)/(F2-D2) = 0.09/0.14 = 64.3%

% Carbonates (See Heiri 2001)= (G2-H2)***1.36**/(F2-D2) = 0.03***1.36**/0.14 = 29.1%