Soil Inorganic Carbon Removal for Organic Carbon Determination on EA/IRMS

Created: DPH
Updated: 5 November 2015 -EPM

Introduction

Soil organic carbon (SOC) is a critical soil property for understanding ecosystem function. Soil carbon represents stored energy, introduced to the system by primary production, and used by heterotrophic organisms. The quantity of C in the soil relative to other essential elements such as nitrogen and phosphorous can indicate the health and fertility of the soil, and is important to understanding the global C cycle and potential ramifications of changing climate. However, in most soils total carbon content includes some fraction of inorganic carbon (IC), which is not biologically available and therefore not often of interest to ecologists examining soil C controls and feedbacks on ecosystem function. At the very least, IC can interfere with the interpretation of the SOC signal. Therefore, IC must be removed prior to SOC determination or quantified and SOC determined by difference from the total C content. In some cases, SOC by difference is not suitable, as when examining the isotopic composition of the soil C, here the IC must be removed so the isotopic signature is representative of the SOC only.

There are several different methods for removing SIC, all have strengths and weaknesses. Described here is a soil HCl rinsing method for soils with ≥ 0.5% IC. Soils with only minimal IC content may do better using the HCl fumigation method detailed elsewhere. In the rinsing method, soils are ground and then treated with a dilute HCl solution to remove all the IC as CO_2. The soils are then rinsed to remove the chloride, which can interfere with the mass spectrometer. The rinsing method is best for research questions involving C only; if total nitrogen (N) concentration or isotopic composition is important the optimal practice is to run a separate set of untreated soil samples. If you are working with dryland soils with low total and available N concentrations, it may be suitable to examine rinsed soils for both C and N concentrations and isotopic values but controls should be run to verify this beforehand.

Materials

Centrifuge tubes, 50 mL (preferably with conical bottom vs. with square foot)
Tube racks (especially for conical bottoms)
HCl solution (5% v/v)
Coarse balance (0.01 g)
Kimwipes and isopropyl alcohol for cleaning spatula and working surface
Centrifuge with 50 mL tube crucibles and 3600 rpm capability
Vortex machine
Carboy or water source of Nanopure water
Graduated cylinder or bottle top pipette, 50 mL
Oven @ 50°C for 24 hours
Scintillation vials or other archiving container

Procedures

Treatment Procedure:

1. Label centrifuge tubes with soil ID #. Weigh 1.0 g ± 0.1 of ground soil into respective tubes. There is no need to record weights.

2. Add 10 mL of 5% v/v HCl to each tube and swirl to mix. Place caps on tubes loosely to allow the evolved CO_2 to escape. Place tube in rack on shaker table and shake overnight (~12 hours).

Rinsing Procedure:

Because the HCl must be removed from the soils, the supernatant must be decanted and the soils rinsed with Nanopure water. However, simply decanting the supernatant or rinse solution will result in the loss of a significant fraction of suspended clay and bias your C and N concentrations and isotopic values. Therefore the soil in solution is settled using a centrifuge prior to decanting. Again, this does little to protect your samples from losses of dissolved C and N species. In general, a bright green HCl solution indicates the loss of inorganic N.

3. Place tubes in centrifuge making sure to balance the tubes evenly between crucibles. For most centrifuges, you will be able to spin down 24 samples at a time (~1 rack of tubes). ***Run centrifuge @ 3600 rpm for 10-15 minutes (depending on soil texture)***.
4. Decant the HCl solution into a waste container (dispose of properly). Be aware of any loose material in solution, it should be free of clay. Occasionally there will be suspended organic material which if decanted is fine (although if soils are ground this should be minimal).If running multiple racks of samples, place next set of samples in centrifuge before step 5.
5. Add 40 mL of Nanopure water to each tube, cap tightly, and vortex until the soil pellet is dispersed and the solution is thoroughly mixed (10-15 seconds each).
6. Repeat step 3, then decant solution into empty container. Again, the solution should be free of clay.
7. Repeat steps 5 and 6 once more (total of 2 rinses).
8. Place tubes in oven @ 50°C for 24-48 hours until dry, then transfer treated soils to scintillation vials or other archiving container. Soils are now ready for packing into tins for elemental and isotopic analysis.

References

Brodie, C. R., Leng, M. J., Casford, J. S., Kendrick, C. P., Lloyd, J. M., Yongqiang, Z., & Bird, M. I. (2011). Evidence for bias in C and N concentrations and δ< sup> 13</sup> C composition of terrestrial and aquatic organic materials due to pre-analysis acid preparation methods. *Chemical Geology*,*282*(3), 67-83.

Robertson, G. P., Coleman, D. C., Bledsoe, C. S., & Sollins, P. (1999). *Standard soil methods for long-term ecological research*. Pg. 89-105. Oxford University Press.