Inorganic Carbon Analysis Using the Modified Pressure-Calcimeter Method

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Introduction

Soil inorganic carbon (SIC) determination is typically performed as a correction to total soil carbon (TOC) content to determine by difference the amount of organic carbon present in the soil (SOC). Otherwise, SIC analysis is typically limited to soils where salinity is a concern and threatens crop productivity or soil health. Nonetheless SIC has been recognized as a potentially important terrestrial C storage pool with different controls and residence times than SOC. Whether determining SIC or SOC, determination by difference is a time consuming and expensive method since you have to run duplicates of every sample.

Alternatively, the modified pressure calcimeter method represents a direct method for SIC determination versus by difference and takes considerably less time and money to perform with comparable limits of detection and accuracy. Using this method, the soil sample is reacted with concentrated HCl in a sealed bottle. This reaction produces CO2 which creates pressure in the bottle. The bottle is then punctured with a needle connected to a pressure transducer and a voltage meter. The pressure released from the bottle is measured as a voltage and related to a standard curve to determine the percent SIC.

Equipment needed

Pressure transducer (Setra model 280, 0-15 PSIG, 0.03-5.03 VDC output)
Power supply (DC 24V)
Digital volt meter
Tygon tubing ¼" ID (25-cm in length)
18-gauge Luer-Lok hypodermic needle
0.6 um particle filter (Leco 768-980)
20 mL and 100mL Wheaton serum bottles (no. 223766)
Wheaton rubber stoppers, (no. 224100-192)
20 mm Wheaton Aluminum caps (no. 224193-06)
20 mm Wheaton crimper
0.5 dram vials
Reagent: 6M HCl with 3% FeCl2
Calcium Carbonate
Silica Flour or silica sand

Standards

One standard curve should be run each day with each batch of samples. Use the appropriate standard range for the sensitivity desired:

- Using reagent grade CaCO3 and Silica flour or ground sand, prepare standards with 0.06, 0.12, 0.24, 0.6, 1.2, 1.8, 3.6, 6, and 12% ***CaCO3*** in 20mL bottles.
- If using the 100mL bottles, prepare standards including 1.8, 3.6, 6, 12, 24, 48% ***CaCO3***.

Reagents

6M HCl with 3% by weight $FeCl_2.4H_2O$

a. Using a **1 L** volumetric Pyrex flask, add ~300 ml DI-H2O.
b. In the hood, add **30 g** $FeCl_2.4H_2O$ and swirl to mix.

c. In the hood, slowly add **500 ml** HCl to the 1 L flask (**flask will get hot!**)
d. Allow reagent to cool, then dilute to mark and mix.

Note: When working with acids, always work in the fume hood and wear protective gloves, glasses, and a lab coat. **Also, ALWAYS ADD ACID TO WATER, NOT WATER TO ACID!!!**

Procedure

1. Weigh **1 g** of ground sample/standard into serum bottle, either 20ml or 100ml, depending on the estimated IC percentage and sensitivity desired. Perform this for all samples that will be analyzed for step 2. As long as your samples are less than 1.8% IC, it is probably safe to use the 20ml bottle. With higher concentrations you risk shattering the bottle!
2. Add 2 mL of the HCl reagent to as many 0.5 dram vials as there are samples.
3. Carefully add vial containing HCl reagent to bottle with tweezers making sure the HCl does not react with the soils yet. Periodically rinse tweezers to keep from corroding. Wear gloves, glasses, and lab coat.

   Note: If you are using the threaded cap style vials, the vial opening diameter is sufficiently narrow to allow surface tension to prevent the HCl reagent from spilling out if tipped over in the bottles. In this case, vials can be slipped into the top of the serum bottles by hand.

4. Place septum on bottle top, then aluminum seal. Seal with crimper tool. **Take care not to spill acid!**
5. Once all the bottles are sealed, quickly agitate the bottles to knock over vial so that acid comes in contact with sample. Ensure that acid and sample are well mixed. Allow bottle to sit overnight.
6. Measure voltage of sample as follows:
   a. Turn on power supply to pressure transducer.
   b. Turn on volt meter. Press **range** button until you are in **volts**. Press **max/min** button. Display should indicate **max.** In this mode the maximum voltage reading is displayed and will correspond to the max pressure reading.
   c. Record the "baseline" reading from the volt meter from each sample to subtract out later during your calculations.
   d. Pierce the septa with needle hooked up to pressure transducer. Record voltage displayed on the volt meter.

      Note: After several samples the needle may become clogged with rubber from the septa. If this happens you will need to push it out using a thin wire. In addition, after several samples, the lubricant on the needle will wear off making it difficult to insert the needle; if you notice extra resistance when piercing the septa rub a very small amount of vacuum grease on the needle every 4-8 samples.

7. Calculate a linear regression for the calibration curve that uses $CaCO_3$. Use the following formula to calculate % SIC (also referred to as total inorganic carbon, TIC).

$$\%TIC = \%\ CaCO_3 * 12/100$$

The term 12/100 represents the mole fraction of carbon contained within each molecule of $CaCO_3$.

Additional Notes

The method detection limit (MDL) for the 20 ml bottle is 0.017% IC. For the 100 ml bottle the MDL is 0.042% IC.

References

L.A Sherrod, G. Dunn, G.A. Peterson, R.L. Kolberg. 2002. Inorganic carbon analysis by modified pressure-calcimeter method. Soil Sci. Soc. Am. J. 66:299-305.

Sparks, D. L., Page, A. L., Helmke, P. A., Loeppert, R. H., Soltanpour, P. N., Tabatabai, M. A., ... & Sumner, M. E. (1996). *Methods of soil analysis. Part 3-Chemical methods*. Pg. 437-474. Soil Science Society of America Inc..