

Modified Pressure Calcimeter Method

Method based on: L. A. Sherrod, G. Dunn, G. A. Peterson, and R. L. Kolberg. 2002. Inorganic Carbon Analysis by Modified Pressure-Calcimeter Method. *Soil Sci. Soc. Am. J.* 66: 299–305.

Overview:

Soil sample is reacted with HCl in a sealed container. 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 volt meter. The pressure released from the bottle is measured as a voltage.

Materials:

Pressure transducer (Setra model 280, 0-15 PSIG, 0.03-5.03 VDC output)
Power supply (IEI model: PIP 1324 transformer, Input: AC 120V 60Hz, Output: DC 24V 1000 mA)
Digital multimeter (Fisher Scientific)
14-gauge copper wire to connect pressure transducer to power supply
25-cm of Nalgene Brand Tygon tubing ¼" ID
¼" Swagelok tubing adapter
Teflon tape
Adhesive or sealant (Goop brand works well)
18-gauge Luer-Lok hypodermic needle
0.6µm particle filter (Leco 768-980)
20mL and 100mL Wheaton serum bottles (no. 223766)
Wheaton rubber stoppers, (no. 224100-192)
20mm Wheaton Aluminum caps (no. 224193-06)
20mm Wheaton crimper
2mL auto sampler vials
6M HCl with 3% FeCl₂
Calcium Carbonate
Silica Flour or silica sand

Making Standards:

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. Standards should be made for each individual run, as it is difficult to get a good calibration curve from prepared batches of standard.

Make 6M HCl with 3% FeCl₂. In a 200mL beaker weigh out 6g of:4H₂O. In same beaker mix 100mL of concentrated HCL with 100mL of DI water. ****Always add acid to water and never the other way around****

Procedure:

1. Make sure pressure calcimeter and voltage meter are connected properly to the power supply. Connect either side of filter to Tygon Tubing and create a gas tight connection with the sealant. Connect tubing to the pressure transducer using the Swagelok tubing adapter. Wrap

adapter threads with Teflon tape before screwing into the transducer. Connect the needle to the filter using the remaining tubing.

2. Soils with less than 12% CaCO₃:
 - a. Before running each sample set run a blank and a calibration curve. It is important that the calibration curve and samples are prepared at the same time.
 - b. Add 1.00g of sieved (2mm x 2mm), split, and ground sample (mortar and pestle) to a 20mL serum bottle. Sample can be directly added to a tared bottle. Record weight.
 - c. Pipette 2mL of 6 M HCl solution into the 2mL vial
 - d. Using plastic forceps carefully place the 2mL vial into the serum bottle without spilling **ANY** acid.
 - e. Cap the bottle with a rubber stopper. Seal and crimp the aluminum cap on using the hand crimper.
 - f. Shake the bottle so that all of the HCl reacts with the entire sample
 - g. Let the bottle sit for 5-6 hours
 - h. Turn on the pressure transducer
 - i. Before reading each sample record the “baseline” pressure that is read on the volt meter
 - j. Remove the tear-off cap and puncture the stopper with the hypodermic needle
 - k. Record the voltage after 3-5 seconds
 - l. Make sure to not move the needle once it has pierced the rubber stopper as this can cause pressure to be released from the bottle

3. Soils with greater than 12% CaCO₃
 - a. Follow steps b(i)-b(v) from above using the larger 100mL bottles
 - b. If soils are expected to have more than 50% CaCO₃, consider cutting down the sample mass. Be cautious of samples with high CaCO₃ as the rubber caps may blow off if the pressure is great enough
 - c. React the acid and soil sample by gently swirling the bottle on a flat surface. Avoid getting sample on the side of bottles that has not reacted with acid.
 - d. Follow steps from above.

Calculations:

1. Calculate a linear regression for the calibration curve. Use the following formula to calculate % CaCO₃
$$\% \text{TIC} = \% \text{CaCO}_3 * 12/100$$

2. It may be helpful to subtract out the “baseline” reading from the voltage meter from each sample.