

## **DETERMINATION OF TOTAL CARBON, TOTAL ORGANIC CARBON AND INORGANIC CARBON IN SEDIMENTS**

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### **ABSTRACT**

Total carbon content is determined in dried sediments and total organic carbon is determined in dried and acidified sediments using a LECO CR-412 Carbon Analyzer. Sediment is combusted in an oxygen atmosphere and any carbon present is converted to CO<sub>2</sub>. The sample gas flows into a non-dispersive infrared (NDIR) detection cell. The NDIR measures the mass of CO<sub>2</sub> present. The mass is converted to percent carbon based on the dry sample weight. The total organic carbon content is subtracted from the total carbon content to determine the total inorganic carbon content of a given sample.

### **1.0 INTRODUCTION**

Total carbon (TC) includes both inorganic and inorganic sample constituents. Total organic carbon (TOC) is determined by treating an aliquot of dried sample with sufficient phosphoric acid (1:1) to remove inorganic carbon prior to instrument analysis. Percent TOC and TC are determined in sediments dried at 105°C using a LECO CR-412 Carbon Analyzer. Prepared sediments are combusted at 1,350°C in an oxygen atmosphere using a LECO CR-412. Carbon is oxidized to form CO<sub>2</sub>. The gaseous phase flows through two scrubber tubes. The first scrubber tube is packed with Drierite® (CaSO<sub>4</sub>) and copper granules to trap water and chlorine gas; the second scrubber tube is packed with Anhydron® (Mg(ClO<sub>4</sub>)<sub>2</sub>) to remove residual moisture. The gaseous phase then flows through a non-dispersive infrared (NDIR) detection cell tuned to selectively respond to CO<sub>2</sub>. The integrated area under the signal detected is proportional to the amount of CO<sub>2</sub> passing through the NDIR cell. The weight-corrected result is % C.

## **2.0 APPARATUS AND MATERIALS**

### **2.1 EQUIPMENT**

- Conditioning oven, electric, gravity convection, capable of maintaining a stable temperature of up to 200°C
- Combustion furnace, electric, capable of combusting glassware at 400°C for at least 4 hours
- LECO CR-412 Carbon Analyzer, IR detector and 36 position autosampler rack
- Glazed and unglazed combustion boats
- Analytical balance, capable of weighing to 1 mg
- Calibrated weights, certified
- Glass measuring scoop
- Mortar and pestle
- Aluminum weighing boats
- Forceps
- Glass wool

### **2.2 REAGENTS**

- Phosphoric acid, 1:1 v/v
- Calcium carbonate, 99% purity
- Anhydron®
- Drierite®
- Granular copper, 20-30 mesh
- Oxygen, 99% purity

## **3.0 PROCEDURE**

All glassware and ceramic ware used in sample processing are combusted at 400°C for at least 4 hours. Samples remain frozen at -20°C until processing. Sediment samples are thawed and homogenized. The sample is dried in an oven at 40°C. A portion of sample is removed, ground and homogenized. An aliquot of dried, homogenized sample is placed in an aluminum-weighing pan and dried at 105°C.

The LECO CR-412 Carbon Analyzer is calibrated prior to the analysis of samples. Different amounts of high purity calcium carbonate standard (99.95% purity, carbon content of 12.0%) are used to calibrate the instrument. The approximate amounts of calcium carbonate used for the six-point calibration are; 0.01 g, 0.05 g, 0.10 g, 0.25 g and 0.50 g. An empty carbon-free combustion boat is analyzed as a blank for the calibration curve. The calibration curve provides an analysis range of approximately 0.0 to 0.06 g

total carbon. Each calibration standard must fall within 3% of the known percent carbon value to meet acceptance criteria. A continuing calibration check standard (mid-level standard) is analyzed every ten samples and must be within 5% of the known value of the standard.

Total carbon is analyzed by placing approximately 0.350 g of dried, ground and homogenized sample into a clean, carbon-free combustion boat. The sample boat is placed on the autosampler rack assembly and loaded onto the LECO carbon analyzer.

Total organic carbon is analyzed by placing approximately 0.350 g of dried, ground and homogenized sample into a clean, carbon-free combustion boat. Each sample boat is treated with phosphoric acid drop by drop until the sample stops “bubbling” and the sample is completely moist with acid. The sample is placed into an oven set at 40°C for 24 hours and then transferred to an oven set at 105°C. Once the sample is dry, the boat is placed on the autosampler rack assembly and loaded onto the LECO carbon analyzer.

#### **4.0 QUALITY CONTROL (QC)**

All reagents used are verified to be contaminant free. All equipment and glassware used to analyze samples are verified to be carbon-free or are combusted at 400°C for a minimum of 4 hours. The calibration and accuracy of balances, weights, pipettors and thermometers are checked daily. The calibration and accuracy of balances, weight, pipettors and thermometers are verified yearly by an independent source. All samples are shipped and received under chain-of-custody. A series of quality control samples are processed with each batch of 20 samples or less. The following quality controls are used to ensure the accuracy and precision of data.

- Method Blank. Method blanks are clean, carbon-free combustion boats. A method blank is analyzed with each batch of 20 or fewer samples. The method blank is analyzed in a manner identical to samples.
- Laboratory Duplicates. A sample is analyzed in duplicate with each analytical batch of 20 or fewer samples.
- Standard Reference Material (SRM). ASRM from the National Institute of Standards and Technology (NIST) is analyzed with each analytical batch of 20 or fewer samples. SRMs are analyzed in a manner identical to samples, except they are not acidified for TOC analysis. The SRM is only certified for TC and is not certified for TOC. However, an acidified SRM is analyzed in each TOC analytical batch and used as a laboratory control sample (LCS).

## 5.0 CALCULATIONS

### 5.1 CARBON CONTENT (MG)

$$\text{Carbon (g)} = (b)(A) + a$$

Where:

b = the slope of the linear calibration curve (g per unit area)

A = the area under the sample curve

a = the intercept of the calibration curve (g)

Note: When samples have been acidified, Organic Carbon (g), replaces Carbon (g) in the above equation.

### 5.2 PERCENT TOTAL CARBON (TC)

$$\text{TC (\%)} = \frac{\text{Carbon (g)}}{W \text{ (g)}}$$

Where:

W (g) = dry sediment analysis weight (g)

### 5.3 PERCENT TOTAL ORGANIC CARBON CONTENT (TOC)

$$\text{TOC (\%)} = \frac{\text{Organic Carbon (g)}}{W \text{ (g)}}$$

### 5.4 PERCENT TOTAL INORGANIC CARBON CONTENT (TIC)

$$\text{TIC (\%)} = \text{TC (\%)} - \text{TOC (\%)}$$

To express TIC as a percent calcium carbonate (CaCO<sub>3</sub>), use the following equation.

$$\text{CaCO}_3 \text{ (\%)} = (\text{TC} - \text{TOC}) \times 8.33$$

**5.5 DUPLICATE SAMPLE ANALYSES**

$$\text{RPD} = \frac{|(\text{Carbon}_{\text{sample1}} - \text{Carbon}_{\text{sample2}})|}{\left(\frac{\text{Carbon}_{\text{sample1}} + \text{Carbon}_{\text{sample2}}}{2}\right)} \times 100$$