

## Instrument: 832 Series

# Determination of Total Carbon (TC) and Total Organic Carbon (TOC) in Soil by Acid Digestion and Combustion

LECO Corporation; Saint Joseph, Michigan USA



### Introduction

Total organic carbon (TOC) determination in soil is a widely used method for evaluating soil quality in agriculture. TOC levels are a key indicator of soil vitality, fertility, and capacity for atmospheric carbon sequestration. TOC represents the carbon stored in soil organic matter, which plays a vital role in supporting microbial life, enhancing nutrient availability, and improving soil structure. These benefits translate directly into better water retention, reduced erosion, and more resilient crops—key factors for regenerative systems focused on ecosystem restoration and productivity. TOC levels can also be used to monitor the effectiveness of sustainable practices like crop rotation, cover cropping, reduced tillage, and the addition of organic matter such as manure and compost. Measuring soil TOC levels helps growers optimize the use of soil amendments like compost and manure to increase organic matter without over-application.

### Instrument Model and Configuration

The LECO C832 is a carbon determinator that utilizes high-temperature combustion in a whole oxygen environment to analyze a wide range of sample types. It features a ceramic combustion furnace in a horizontal orientation designed to analyze macro sample masses. Analysis begins when a sample is weighed into a ceramic combustion boat and inserted into the furnace where combustion is initiated under a controlled flow of oxygen. After a preset time, additional oxygen is applied directly above the sample using a ceramic lance to accelerate and complete the combustion process. The gases are swept from the furnace and passed through anhydrous for the removal of moisture and are then carried to a non-dispersive infrared (NDIR) cell for detection of carbon as CO<sub>2</sub>.

### Method Reference

ISO 10694: Soil Quality - Determination of Organic and Total Carbon after Dry Combustion (Elementary Analysis)

### Method Summary

TOC determination is typically performed by treating the sample with acid to remove the inorganic carbonate species by converting and releasing the carbonate components as carbon dioxide. This procedure is referred to as the acid digestion method. Documented methods, such as ISO 10694, recommend the use of dilute hydrochloric acid (HCl) for the removal of carbonates, which effectively removes carbonates without significantly affecting organic carbon, preserving the TOC content. Alternatively, sulfurous acid (H<sub>2</sub>SO<sub>3</sub>) may also be used to treat soil samples. Although sulfurous acid is weaker than HCl, it still effectively removes carbonate carbon from soil

with sufficient treatments. The acid-treated sample can then be analyzed to determine the TOC content, a method often referred to as the non-carbonate carbon method for estimating TOC content.

A weighed sample is treated with dilute acid to remove carbonate carbon and then dried in an oven to remove excess acid. The dried acid-treated sample is then analyzed to determine the non-carbonate carbon content of the material, which is usually referred to as the total organic carbon (TOC) content. A separate portion of a non-acid-treated sample can be analyzed to determine the total carbon content of the material. Obtaining a total carbon value allows for the inorganic carbon content of the material to be calculated by difference. The difference between the total carbon results and the acid-treated (TOC) results is referred to as the carbonate carbon content or the total inorganic carbon (TIC) content.

Two different methods were utilized to generate data for this application note. The first method involved the use of dilute hydrochloric acid to perform the acid digestion step. The second method involved the use of sulfurous acid to perform the acid digestion step.

### Method 1: Acid Digestion Using Hydrochloric Acid (HCl)

When hydrochloric acid is used for acid digestion, residual chloride in the sample volatilizes during combustion. As chloride gas is highly corrosive, this poses a risk of damage to downstream instrument components. To mitigate this, a halogen (chlorine) trap was installed on the instrument in the secondary reagent tube (additional details regarding the installation of a halogen trap can be found in step 2 of the "Procedure for TOC Determination"). Additionally, to prevent loss of sample due to strong effervescence, a dilute solution of HCl (1:11 HCl:H<sub>2</sub>O (1 N)) was used for the first two soil treatments. The first two soil treatments were then followed by a third treatment using a more concentrated solution of HCl (1:3 HCl:H<sub>2</sub>O (3 N)) to ensure complete digestion of the carbonates in the sample.

### Method 2: Acid Digestion Using Sulfurous Acid (H<sub>2</sub>SO<sub>3</sub>)

When sulfurous acid is used to perform the acid digestion step, no halogens are present to be released during combustion, eliminating the need for a halogen (chlorine) trap and the associated costs of additional reagents and consumables.

Sulfurous acid is also inherently less corrosive than hydrochloric acid, reducing the risk of damage to instrument components. Additionally, the same acid solution can be used for all of the treatments, eliminating the need to prepare a second, more dilute solution. However, more treatments are typically required when using sulfurous acid than when using HCl to fully remove carbonates.

### Sample Preparation

A representative, uniform sample is required. Samples should be ground to a fine powder. Samples should be dried prior to analysis or corrected for moisture (see note below). Samples for TOC determination should be treated with dilute acid following the procedure outlined in the "Acid Treatment for TOC Determination" procedure section in the "TOC Determination" portion of the procedure. Separate portions of the sample material should be used for total carbon and TOC determination. Reference materials should be prepared according to the instructions on the certificate.

*Note: Expressing carbon content on a dry basis is a crucial factor in reporting accurate carbon results. It is recommended that samples for total carbon determination either be dried in an oven for 1 hour at 105 °C prior to weighing and analysis, or a moisture content be determined on the day of analysis, which can be used for a dry basis correction of the carbon results using the instrument's software. It is recommended that samples for TOC determination be dried in an oven for 1 hour at 105 °C prior to weighing and acid treatment.*

### Accessories

#### Total Carbon Determination

528-203 Ceramic Combustion Boats\* or 528-206 Non-Porous Ceramic Combustion Boats\*, 761-929 Crucible Tongs, and 501-614 Spatula

#### Non-Carbonate (TOC) Determination

528-206 Non-Porous Ceramic Combustion Boats\*, 202-001-315 Halogen Trap Kit, a 1.2:100 solution of 501-179 LECONAL (for use on hydrophobic samples), a 1:11 HCl:H<sub>2</sub>O (1 N) solution and a 1:3 HCl:H<sub>2</sub>O (3 N) solution, or a 6 % sulfurous acid solution, temperature-controlled oven, 761-929 Crucible Tongs, 501-614 Spatula, and a graduated pipette

*\*For optimal precision, combustion boats should be baked in a muffle furnace at 1,000 °C for a minimum of 1 hour prior to use. Once the combustion boats have cooled, they should be transferred to a desiccator for storage until use. If the combustion boats are not used within twenty-four hours, they should be re-baked. After baking, handle combustion boats with clean tongs only; do not use fingers.*

*Note: The 528-206 Non-Porous Ceramic Combustion Boats are specifically designed so that acid treatment can be performed directly in the boat. These boats may be reused for multiple analyses; however, they should be visually inspected for any signs of damage or cracking prior to each reuse, and if any damage or cracking is observed, they should be discarded. When using an autoloader, LECO recommends lining the boat bucket with ~ 60.0 g of 501-608 Quartz Wool to cushion the impact of the boat when it drops in to the bucket in order to extend boat lifetime*

### Reference Materials

LCRM®, LRM®, NIST, or other suitable reference materials.

### General

Furnace Temperature	1350 °C
Lance On Delay	20 s
Manual Loading Model	Single Sample
Nominal Mass	1.0000 g
Sample Cool Time	15 s (Autoloaders)

### Element Parameters

Wait for Baseline Stability	Yes
Starting Baseline	2 s
Use Comparator	Yes
Comparator Level	0.30 %
Minimum Integration Time	90 s
Maximum Integration Time	360 s
Auto Detect Data Missed Time	3 s
Integration Delay	0 s

*Note: Refer to 832 Series Operator's Instruction Manual for Parameter definitions.*

### Procedure for Total Carbon Determination

- Prepare instrument for operation as outlined in the operator's instruction manual.
- Condition the system.
  - From the Analysis Screen, use the Login Bar to add three sample replicates.
  - Weigh ~0.25 g of calcium carbonate (or soil sample) into a pre-baked 528-203 Ceramic Combustion Boat.
  - Enter sample mass and identification into the Analysis Screen.
  - Place the ceramic combustion boat containing the sample in front of the furnace entrance (for manual loading systems), or into the appropriate position in the autoloader.
  - For manual loading systems, initiate the analysis sequence, and when prompted by the software, load the ceramic combustion boat containing the sample into the furnace and press the Analyze button.
  - Perform steps 2b through 2d (or 2b through 2e for manual loading systems) a minimum of three times.
  - For auto-loading systems, initiate the analysis sequence.
- Determine the instrument blank.
  - From the Analysis Screen, use the Login Bar to add five or more blank replicates.
  - Place a pre-baked 528-203 Ceramic Combustion Boat in front of the furnace entrance (for manual loading systems) or place the required number of pre-baked ceramic combustion boats (minimum of five) into the appropriate positions in the autoloader.
  - For manual loading systems, initiate the analysis sequence, and when prompted by the software, load the ceramic combustion boat into the furnace and press the Analyze button.
  - For manual loading systems, perform steps 3b through 3c a minimum of five times.
  - For auto-loading systems, initiate the analysis sequence.
  - Set the blank following the procedure outlined in the operator's instruction manual.
- Calibrate or drift correct.
  - From the Analysis Screen, use the Login Bar to add the desired number of calibration/drift replicates (minimum of three).

*Note: The standard deviation of the last three blanks should be less than or equal to 0.001 % C (10 ppm). Additional blanks beyond the recommended five may be required in order to achieve the recommended precision.*

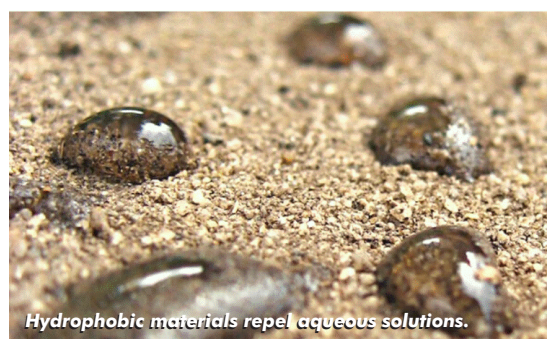
- b. Weigh an appropriate mass of a suitable reference material into a pre-baked 528-203 Ceramic Combustion Boat and spread the material evenly within the combustion boat.
  - c. Enter sample mass and identification into the Analysis Screen.
  - d. Place the ceramic combustion boat containing the reference material in front of the furnace entrance (for manual loading systems), or into the appropriate position in the autoloader.
  - e. For manual loading systems, initiate the analysis sequence, and when prompted by the software, load the ceramic combustion boat containing the sample into the furnace and press the Analyze button.
  - f. Perform steps 4b through 4d (or 4b through 4e for manual loading systems) a minimum of three times for each calibration/drift sample used.
  - g. For auto-loading systems, initiate the analysis sequence.
  - h. Calibrate or drift correct the instrument following the procedure outlined in the operator's instruction manual.
  - i. Verify the calibration/drift correction by analyzing an appropriate mass of another/different suitable reference material, and confirm that the results are within the acceptable tolerance range.
5. Analyze the samples.
    - a. From the Analysis Screen, use the Login Bar to add the desired number of sample replicates.
    - b. Weigh ~0.25 g to ~0.5 g of the sample into a pre-baked 528-203 Ceramic Combustion Boat and spread the sample evenly within the combustion boat.
    - c. Enter sample mass and identification into the Analysis Screen.
    - d. Place the ceramic combustion boat containing the sample in front of the furnace entrance (for manual loading systems), or into the appropriate position in the autoloader.
    - e. For manual loading systems, initiate the analysis sequence, and when prompted by the software, load the ceramic combustion boat containing the sample into the furnace and press the Analyze button.
    - f. Perform steps 5b through 5d (or 5b through 5e for manual loading systems) for each sample to be analyzed.
    - g. For auto-loading systems, initiate the analysis sequence.

## Procedure for TOC (Non-Carbonate Carbon) Determination

### Sample Preparation for TOC Determination

Note: Some materials are hydrophilic and readily absorb the acid solution, while others are hydrophobic and repel the acid solution, preventing it from soaking into the sample. If the acid solution beads on the sample rather than soaking in, the sample is hydrophobic (see images below). It is important that the acid solution soaks into the material so that it can react with the carbonate carbon within the sample. To allow for the absorption of the acid solution into hydrophobic materials, a surfactant is added to the sample prior to acid treatment. The procedure below should be used to make a surfactant solution, if needed. Add ~0.1 mL of the surfactant solution to the hydrophobic samples before acid treating the samples and acid blank samples. Do not add additional surfactant solution during subsequent acid addition steps. If a material is hydrophobic and

requires treatment with a surfactant solution, the surfactant should be used for acid blank determination as well. If analyzing a hydrophobic sample, surfactant should be added to the combustion boat prior to adding the acid when determining the acid blank.



### Procedure for Preparing Surfactant Solution for the Treatment of Hydrophobic Samples

1. Using a graduated pipette, add 0.1 mL of LECO 502-889 LECONAL™ concentrate to a clean 100 mL volumetric flask and dilute to volume using deionized water. Mix thoroughly.
2. Using a fresh graduated pipette, add 1.2 mL of the prepared dilute LECONAL solution to a separate, clean 100 mL volumetric flask and dilute to volume using deionized water. Mix thoroughly prior to use.

### Method 1: Acid Treatment Procedure Using Hydrochloric Acid

1. Weigh ~0.25 g to ~0.5 g of the dried sample into a pre-baked 528-206 Non-Porous Ceramic Combustion Boat and spread the sample evenly within the combustion boat. Record the mass (This is the mass that will be entered into the instrument Analysis Screen for analysis of the sample following acid treatment).
2. Using a graduated pipette, add ~1.0 mL of a 1:11 HCl:H<sub>2</sub>O (1 N) solution to completely wet the sample. The sample will effervesce, indicating a reaction between the acid solution and the carbonates within the sample. If the acid solution beads on the surface of the sample rather than soaking in, refer to the Note below.
3. Dry the acid-treated sample in the combustion boat in a drying oven (~105 °C) for one hour, or until dry. Allow the crucible containing the sample to cool for at least 10 minutes prior to adding additional acid solution.

4. Perform steps 2 through 3 two times.
5. Perform a third acid treatment using a 1:3 HCl:H<sub>2</sub>O (3 N) solution. Using a fresh graduated pipette, add ~1.0 mL of a 1:3 HCl:H<sub>2</sub>O (3 N) solution to completely wet the sample.
6. Dry the acid-treated sample in the combustion boat in a drying oven (~105 °C) for one hour, or until dry.

#### **Method 2: Acid Treatment Procedure Using Sulfurous Acid**

1. Weigh ~0.25 g to ~0.5 g of the dried sample into a pre-baked 528-206 Non-Porous Ceramic Combustion Boat and spread the sample evenly within the combustion boat. Record the mass (This is the mass that will be entered into the instrument Analysis Screen for analysis of the sample following acid treatment).
2. Using a graduated pipette, add ~1.0 mL of a 6 % sulfurous acid solution to completely wet the sample. The sample will effervesce, indicating a reaction between the acid solution and the carbonates within the sample. If the acid solution beads on the surface of the sample rather than soaking in, refer to the Note below.
3. Dry the acid-treated sample in the combustion boat in a drying oven (~105 °C) for one hour, or until dry. Allow the crucible containing the sample to cool for at least 10 minutes prior to adding additional acid solution.
4. Perform steps 2 through 3 a total of seven times, or until no additional effervescing occurs (Typically a minimum of seven treatments is required when using sulfurous acid for successful removal of carbonates).

#### **Sample Preparation for Acid Blank Determination and Carbonate Removal Verification**

*Note: The same acid solution that was used to treat the samples should be used for acid blank determination. If a surfactant was used for hydrophobic soil samples, the same volume of surfactant solution should be added to the combustion boat along with the acid solution for acid blank determination. Do not add additional surfactant solution during subsequent acid addition steps.*

#### **Acid Blank Determination Procedures**

##### **Method 1: Acid Blank Determination Procedure for Samples Treated with Hydrochloric Acid**

1. Prepare samples for acid blank determination.
  - a. Using a graduated pipette, add ~1.0 mL of a 1:11 HCl:H<sub>2</sub>O (1 N) solution into a pre-baked 528-206 Non-Porous Ceramic Combustion Boat.
  - b. Dry the acid blank sample in the combustion boat in a drying oven (~105 °C) for one hour, or until dry. Allow the crucible containing the acid solution to cool for at least 10 minutes prior to adding additional acid solution.
  - c. Perform steps 1a through 1b two times.
  - d. Perform a third acid treatment using a 1:3 HCl:H<sub>2</sub>O (3 N) solution. Using a fresh graduated pipette, add ~1.0 mL of a 1:3 HCl:H<sub>2</sub>O (3 N) solution to completely wet the sample.
  - e. Dry the acid blank sample in the combustion boat in a drying oven (~105 °C) for one hour, or until dry.

##### **Method 2: Acid Blank Determination Procedure for Samples Treated with Sulfurous Acid**

1. Prepare samples for acid blank determination.
  - a. Using a graduated pipette, add ~1.0 mL of a 6 % sulfurous acid solution into a pre-baked 528-206 Non-Porous Ceramic Combustion Boat.
  - b. Dry the acid blank sample in the combustion boat in a drying oven (~105 °C) for one hour, or until dry. Allow the crucible containing the acid solution to cool for at least 10 minutes prior to adding additional acid solution.
  - c. Perform steps 1a through 1b the same number of times as the samples were treated.

#### **Carbonate Removal Verification Procedures**

##### **Method 1: Carbonate Removal Verification Procedure for Samples Treated with Hydrochloric Acid**

1. Prepare samples for carbonate removal verification.
  - a. Weigh an equivalent mass<sup>22</sup> of calcium carbonate, or synthetic carbon into a pre-baked 528-206 Non-Porous Ceramic Combustion Boat and spread the sample evenly in the combustion boat. Record the mass (This is the mass that will be entered into the instrument Analysis Screen for analysis of the sample following acid treatment).
  - b. Using a graduated pipette, add ~1.0 mL of a 1:11 HCl:H<sub>2</sub>O (1 N) solution to completely wet the sample. When samples containing high amounts of carbonates are treated, heavy effervescence will occur. Therefore, it is recommended that the acid solution be added slowly to the sample to prevent sample loss.
  - c. Dry the acid-treated sample in the combustion boat in a drying oven (at ~105 °C) for one hour, or until dry. Allow the crucible containing the calcium carbonate or synthetic carbon sample to cool for at least 10 minutes prior to adding additional acid solution.
  - d. Perform steps 2a through 2c two times.
  - e. Perform a third acid treatment using a 1:3 HCl:H<sub>2</sub>O (3 N) solution. Using a fresh graduated pipette, add ~1.0 mL of a 1:3 HCl:H<sub>2</sub>O (3 N) solution to completely wet the sample.
  - f. Dry the acid-treated sample in the combustion boat in a drying oven (at ~105 °C) for one hour, or until dry.

##### **Method 2: Carbonate Removal Verification Procedure for Samples Treated with Sulfurous Acid**

1. Prepare samples for carbonate removal verification.
  - a. Weigh an equivalent mass<sup>22</sup> of calcium carbonate, or synthetic carbon into a pre-baked 528-206 Non-Porous Ceramic Combustion Boat and spread the sample evenly in the combustion boat. Record the mass (This is the mass that will be entered into the instrument Analysis Screen for analysis of the sample following acid treatment).
  - b. Using a graduated pipette, add ~1.0 mL of a 6 % sulfurous acid solution to completely wet the sample. When samples containing high amounts of carbonates are treated, heavy effervescence will occur. Therefore, it is recommended that the acid solution be added slowly to the sample to prevent sample loss.

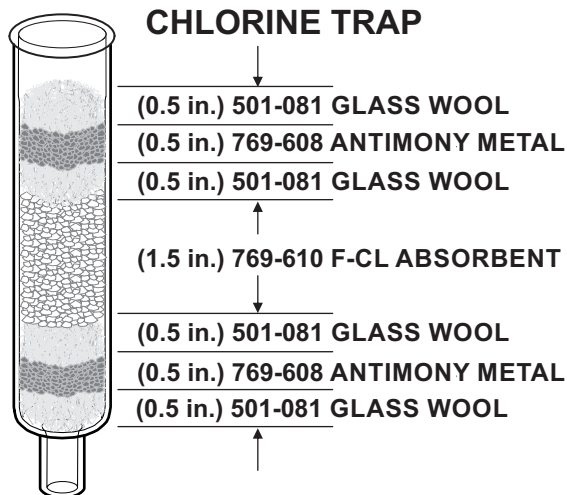
- c. Dry the acid-treated sample in the combustion boat in a drying oven (at ~105 °C) for one hour, or until dry. Allow the crucible containing the calcium carbonate or synthetic carbon sample to cool for at least 10 minutes prior to adding additional acid solution.
- d. Perform steps 2a through 2c the same number of times as the samples were treated.

*"For optimal precision, the mass of calcium carbonate (or synthetic carbon) used to verify carbonate removal should be the same mass as the samples being analyzed.*

*Note: Acid-treated calcium carbonate (or synthetic carbon) samples are analyzed in order to verify that the acid treatment procedure was successful in removing carbonate carbon. Results for the acid-treated calcium carbonate (or synthetic carbon) samples should be < 0.1 % C to confirm successful removal of carbonates. The same type, concentration and amount of acid solution, and the same number of treatments that were used to treat the samples, must be used to treat the calcium carbonate samples and the acid blank samples.*

### Analysis Procedure for TOC Determination

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. If hydrochloric acid was used to treat the soil samples, install a halogen (chlorine) trap on the instrument in the secondary reagent tube (refer to LECO Product Information Bulletin No. 202-001-315 Halogen Trap Kit Instructions, and the diagram below). If sulfuric acid was used to treat the soil samples, a halogen trap is not required.



3. Condition the system.
  - a. Follow the same procedure for conditioning the system as outlined in step 2 of the "Total Carbon Determination" section of the procedure.

*Note: Fresh chlorine trap reagents may require conditioning by analyzing one to two acid-treated samples or a material containing an equivalent mass percent of chlorine. The trap has been depleted when the F-Cl Absorbent has turned a dark, brown color and/or the bottom layer of the antimony metal has become tarnished.*

4. Determine instrument blank<sup>†</sup>.
  - a. Follow the same procedure for determining the instrument blank as outlined in step 3 of the "Total Carbon Determination" section of the procedure.

*Note: For optimal precision, pre-baked 528-206 Non-Porous Ceramic Combustion Boats should be used when determining the instrument blank for TOC determination.*
5. Determine acid blank<sup>†</sup>.
  - a. From the Analysis Screen, use the Login Bar to add five or more blank replicates.
  - b. Place a 528-206 Non-Porous Ceramic Combustion Boat containing a dried acid sample (prepared in step 1 of the "Sample Preparation for Acid Blank Determination" procedure) in front of the furnace entrance (for manual loading systems) or place the required number of combustion boats containing dried acid samples into the appropriate positions in the autoloader.
  - c. Enter the appropriate sample identification information into the appropriate fields in the Analysis Screen.
  - d. For manual loading systems, initiate the analysis sequence, and when prompted by the software, load the combustion boat into the furnace and press the Analyze button.
  - e. For manual loading systems, perform steps 5b through 5d a minimum of five times.
  - f. For auto-loading systems, initiate the analysis sequence.
  - g. Set the acid blank following the blank procedure outlined in the operator's instruction manual.

*Note: The standard deviation of the last three blanks should be less than or equal to 0.001 % C (10 ppm). Additional blanks beyond the recommended five may be required in order to achieve the recommended precision.*

<sup>†</sup>*Instrument blanks are used to set the blank for the reference materials, and acid blanks are used to set the blank for the acid-treated samples.*

6. Calibrate or drift correct.
  - a. Follow the same procedure for calibrating the Instrument as outlined in step 4 of the "Total Carbon Determination" section of the procedure.

*Note: For optimal precision, pre-baked 528-206 Non-Porous Ceramic Combustion Boats should be used when calibrating the instrument for TOC determination.*
7. Analyze acid-treated calcium carbonate (or synthetic carbon) samples for verification of carbonate removal.
  - a. From the Analysis Screen, use the Login Bar to add the desired number (minimum of three) of carbonate removal verification replicates.
  - b. Place a 528-206 Non-Porous Ceramic Combustion Boats containing an acid-treated calcium carbonate (or synthetic carbon) sample (prepared in step 2 of the "Sample Preparation for Carbonate Removal Verification" procedure) in front of the furnace entrance (for manual loading systems) or place the required number of combustion boats containing acid-treated calcium carbonate samples into the appropriate positions in the autoloader.
  - c. Enter the recorded mass of the untreated sample (mass of the sample prior to the acid treatment procedure) and the sample identification information into the appropriate fields in the Analysis Screen.

- d. For manual loading systems, initiate the analysis sequence, and when prompted by the software, load the ceramic combustion boat containing the sample into the furnace and press the Analyze button.
- e. For manual loading systems, perform steps 7b through 7d for each sample to be analyzed.
- f. For auto-loading systems, initiate the analysis sequence.

*Note: Results for the acid-treated calcium carbonate (or synthetic carbon) samples should be < 0.1 % C to confirm successful removal of carbonates.*

8. Analyze the samples.
  - a. From the Analysis Screen, use the Login Bar to add the desired number of sample replicates.
  - b. Place a 528-206 Non-Porous Ceramic Combustion Boat containing an acid-treated soil sample (prepared in the "Acid Treatment for TOC Determination" section of the procedure) in front of the furnace entrance (for manual loading systems) or place the required number of combustion boats containing acid-treated samples into the appropriate positions in the autoloader.
  - c. Enter the recorded mass of the dried, untreated soil sample(s) (mass of the sample after drying and prior to the acid treatment procedure) and the sample identification information into the appropriate fields in the Analysis Screen.
  - d. For manual loading systems, initiate the analysis sequence, and when prompted by the software, load the ceramic combustion boat containing the sample into the furnace and press the Analyze button.
  - e. For manual loading systems, perform steps 8b through 8d for each sample to be analyzed.
  - f. For auto-loading systems, initiate the analysis sequence.

## Typical Results

For soils containing up to 5 % carbon, a linear, force through origin calibration using LECO synthetic carbon reference materials is recommended. For soils containing more than 5 % carbon, a linear, full regression calibration using LECO synthetic carbon and calcium carbonate reference materials is recommended.

Sample	Mass (g)	% TC	HCl Treated Samples		H <sub>2</sub> SO <sub>3</sub> Treated Samples	
			Mass (g)	% TOC	Mass (g)	% TOC
<b>NAPT Soil Sample<sup>††</sup></b>	0.2504	4.05	0.2500	3.18	0.2510	3.10
2022-104	0.2500	3.96	0.2495	3.15	0.2502	3.03
3.92 % ± 0.137 % TC <sup>†</sup>	0.2496	4.00	0.2500	3.08	0.2509	3.00
3.08 % ± 0.215 % TOC <sup>†</sup>	0.2499	3.97	0.2502	3.07	0.2507	3.00
	0.2505	3.97	0.2505	2.98	0.2507	2.98
	$\bar{x}$ =	<b>3.99</b>	$\bar{x}$ =	<b>3.09</b>	$\bar{x}$ =	<b>3.02</b>
	$s$ =	<b>0.04</b>	$s$ =	<b>0.08</b>	$s$ =	<b>0.05</b>
<b>Soil 1</b>	0.2477	3.07	0.2495	2.63	0.2505	2.66
LECO 502-697	0.2515	3.06	0.2510	2.65	0.2500	2.65
Lot 1003	0.2543	3.09	0.2505	2.60	0.2502	2.66
3.05 % ± 0.03 % TC	0.2542	3.06	0.2510	2.61	0.2503	2.66
	0.2543	3.06	0.2499	2.60	0.2506	2.63
	$\bar{x}$ =	<b>3.07</b>	$\bar{x}$ =	<b>2.62</b>	$\bar{x}$ =	<b>2.65</b>
	$s$ =	<b>0.01</b>	$s$ =	<b>0.02</b>	$s$ =	<b>0.01</b>
<b>Soil 2<sup>††</sup></b>	0.2562	13.82	0.2504	13.58	0.2505	13.55
LECO 502-694	0.2534	13.95	0.2498	13.64	0.2508	13.73
Lot 1003	0.2481	13.79	0.2499	13.45	0.2495	13.40
13.61% ± 0.32% TC	0.2483	13.81	0.2494	13.56	0.2492	13.53
	0.2487	13.77	0.2504	13.35	0.2508	13.35
	$\bar{x}$ =	<b>13.83</b>	$\bar{x}$ =	<b>13.51</b>	$\bar{x}$ =	<b>13.51</b>
	$s$ =	<b>0.07</b>	$s$ =	<b>0.11</b>	$s$ =	<b>0.15</b>

$\bar{x}$  = sample mean;  $s$  = sample standard deviation

<sup>††</sup>Soil sample was obtained from the North American Proficiency Testing (NAPT) Program, Soil Science Society of America.

<sup>†</sup>Value was obtained from the North American Proficiency Testing (NAPT) Program, Soil Science Society of America. This value is not a certified value and is an informational value only. The margin of error is a Mean Absolute Deviation (MAD) value.

<sup>‡</sup>Due to the hydrophobic nature of this sample, it was pre-treated with a surfactant to facilitate wetting of the sample prior to the addition of the acid solution.