Application Note

Instrument: 812 Series

EMPOWERING RESULTS
Elemental Analysis | GC Mass Spectrometry | Metallography

Temperature-Dependent Determination of Total Carbon (TC), Total Organic Carbon (TOC), Residual Oxidizable Carbon (ROC), and Total Inorganic Carbon (TIC) in Soil

LECO Corporation; St. Joseph, Michigan USA

Introduction

Total organic carbon (TOC) analysis is a widely used indicator of soil quality in agricultural and ecological systems. Because TOC reflects the amount of carbon stored in soil organic matter, it serves as a strong measure of soil health, fertility, and its capacity to sequester atmospheric carbon. Organic carbon supports essential soil functions, including microbial activity, nutrient cycling, and soil aggregation, which in turn improves water-holding capacity, reduces erosion, and strengthens the resilience of cropping systems. These qualities are especially important in regenerative agriculture, where restoring ecosystem function is a central goal. TOC measurements are also valuable for evaluating the impact of management practices such as crop rotation, cover cropping, reduced tillage, and the application of compost or manure. By tracking TOC levels, growers can optimize soil amendment strategies to build organic matter effectively without excessive application.

Residual oxidizable carbon (ROC) is the portion of soil organic carbon that is more stable than actively cycling carbon, yet can still be oxidized through chemical or thermal processes, typically at temperatures between 400–600 °C. Unlike biologically active carbon pools, ROC represents a long-lasting, non-bioavailable carbon reserve made up of materials such as charcoal, biochar, lignin, humus, and graphite. Because these carbon forms decompose slowly, they contribute to durable soil structure, improved nutrient retention, and gradual carbon release. Measuring ROC provides insight into the stability of soil carbon and helps differentiate persistent carbon from rapidly degradable fractions. By comparing ROC to TOC, growers can estimate how much of the soil organic carbon is readily available for biological processes versus how much of it contributes to long-term carbon sequestration.

Instrument Model and Configuration

The LECO CM812 is a multiphase carbon and moisture determinator with a variable ramp furnace and a wide carbon detection range that is specifically designed to differentiate various forms of carbon by the temperature at which they combust. This capability facilitates the temperature differentiation determination of the different species of carbon in soil.

Method Reference

ISO 17505: Soil and Waste Characterization—Temperature Dependent Differentiation of Total Carbon (TOC400, ROC, TIC900)

Method Summary

Direct measurement of TOC is difficult due to the unique properties of soil. Therefore, analysis must be performed using either an acid digestion method or a temperature-dependent differentiation method. The acid digestion method utilizes a dilute, non-oxidizing acid, such as hydrochloric acid, which is not strong enough to affect organic carbon content. As a result, the TOC content remains intact, while carbonates present in the sample are digested into carbon dioxide gas. The total inorganic carbon (TIC) content can then be calculated by difference as follows:
Total carbon (TC) - TOC = TIC.

The temperature-dependent method requires no sample pretreatment or additional sample preparation, removing hazardous chemicals and time-consuming acid digestion steps from the process. As described in ISO17505, organic species of carbon combust to form CO_2 between 150 °C to 400 °C, residual oxidizable carbon species between 400 °C and 600 °C, and inorganic species thermally decompose between 600 °C and 900 °C, heating to these specific temperatures in an oxidizing environment allows for the differentiation of the TOC content from the ROC and TIC content. The TC content can be calculated as follows: TOC + ROC + TIC = TC. Combined, the temperature-based TOC and ROC contents represent the total non-carbonate carbon content. the total non-carbonate carbon content is equivalent to the TOC content obtained via the acid digestion method described above.

The method utilized to generate data for this application note follows ISO 17505 and involves three ramped steps for the differentiation of the TOC, ROC, and TIC contents. The first step involves ramping the furnace from 150 °C to 400 °C, with a hold time of 240 seconds for the determination of TOC and the second step involves ramping the furnace to 600 °C, with a hold time of 100 seconds for the determination of ROC. The final step involves ramping the furnace to 900 °C for the determination of TIC with a hold time determined by a comparator.

Accessories

781-335 Quartz Boats or 625-505-430 Nickel Boats

Note: The 781-335 Quartz Boats and 625-505-430 Nickel Boats should be pre-baked at 1100 °C, to remove any residual carbon then cooled in a desiccator until time of analysis. If the combustion boats are not used within 24 hours, they should be re-baked. After baking, handle combustion boats with clean tongs only; do not use fingers.

Reference Materials

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

Note: These reference materials require different furnace parameters than those used for total organic carbon determination (refer to Furnace Step Method Parameters listed below).

Sample Preparation

A representative, uniform sample is required. Samples should be dried at 105 °C for 1 hour prior to analysis and stored in a desiccator until use (within 24 hours). Reference materials should be prepared according to the sample preparation statement on the certificate. Please reference the appropriate Safety Data Sheets (SDS) for safe handling of all reference materials and samples.

Method Parameters

Method Parameters: TOC Analysis

General Parameters	
Advanced Gas Flow	No
Purge Flow	4.00 LPM
Purge Time	15 s
Analytical Flow	0.75 LPM
Check Delay	20 s
Nominal Mass	1.0000 g
Manual Loading Mode	Dual Button
Reagent Heater Temperature	High
Afterburner Temperature	850 °C

Element Parameters	*Carbon*
Wait for Baseline Stability	Yes
Integration Delay	0 s
Starting Baseline	2 s
Integrate from Furnace Method	Yes
Use Endline	Yes
Ending Baseline	2 s
Range Select	Auto
Range Lower Limit	800
Range Upper Limit	950

Furnace Parameters: Calibration

Apply Peak Find to This Furnace Method	No

Step 1: Carbon Calibration	
Gas	Oxygen
Peak Monitor	On
Analyte	Carbon
Carbon Threshold Level	0.30
Minimum Time	90 s
Maximum Time	300 s
Mode	Constant
Temperature	1000 °C

Furnace Parameters: TOC, ROC, and TIC Determination

Apply Peak Find to This Furnace Method	Yes

Step 1: TOC Determination Ramping Parameters	
Gas	Oxygen
Peak Monitor	Off
Mode	Ramped
Start Temperature	150 °C
Target Temperature	400 °C
Ramp Rate	70 °C/min

Step 2: TOC Determination Hold Time Parameters	
Gas	Oxygen
Peak Monitor	Off
Mode	Constant
Hold Time	240 s

Step 3: ROC Determination Ramping Parameters	
Gas	Oxygen
Peak Monitor	Off
Mode	Ramped
Target Temperature	600 °C
Ramp Rate	70 °C/min

Step 4: ROC Determination Hold Time Parameters	
Gas	Oxygen
Peak Monitor	Off
Mode	Constant
Hold Time	100 s

Step 5: TIC Determination Ramping Parameters	
Gas	Oxygen
Peak Monitor	Off
Mode	Ramped
Target Temperature	900 °C
Ramp Rate	120 °C/min

Step 6: TIC Determination Hold Time Parameters	
Gas	Oxygen
Peak Monitor	On
Analyte	Carbon
Carbon Threshold Level	0.30
Minimum Time	60 s
Maximum Time	300 s
Mode	Constant

Peak Find Settings

Strategy	User Specified
Peaks	3

Peak 1 - TOC	
Name	TOC
Peak Start	Fixed Time
Start Time	0.4 seconds
Peak End	Fixed Length
Peak Length	511.0 seconds

Peak 2 - ROC	
Name	ROC
Peak Start	End of Previous Peak
Peak End	Fixed Time
End Time	810.0 seconds

Peak 3 - TIC	
Name	TIC
Peak Start	End of Previous Peak
Peak End	End of Sample

Note: The peak start times and peak length settings shown above are recommended starting points. Users may need to make minor adjustments to these parameters to accommodate variations in sample, combustion profile, or peak separation in order to achieve accurate peak integration.

Procedure

1. Prepare the instrument as outlined in the operator's instruction manual.
2. Determine the calibration blank.
    a. From the Analysis Screen, use the Login Bar to add three or more blank replicates.
    b. Select "TOC Analysis" as the Method, and "Calibration" as the Furnace Step Method (parameters noted above).
    c. Place a pre-baked combustion boat on the shelf directly in front of the closed combustion tube door (for manual loading systems) or into the appropriate position in the autoloader.
    d. For manual loading systems, initiate the analysis sequence. When prompted by the software, press the Analyze button and load the combustion boat into the combustion tube until it reaches the sample stop.
    e. For manual loading systems, when analysis is complete, remove the boat and close the combustion tube door.
    f. For manual loading systems, perform steps 2a through 2e a minimum of three times.
    g. For auto-loading systems, perform steps 2a through 2c a minimum of three times, and initiate the analysis sequence.
    h. Set the blank for the reference materials following the procedure outlined in the operator's instruction manual.
3. Calibrate the system.
    a. Weigh ~0.10 to 0.25 g of the selected reference material into a pre-baked combustion boat. Enter reference material mass and identification into the Analysis Screen.
    b. Select "TOC Analysis" as the Method and "Calibration" as the Furnace Step Method (parameters noted above).
    c. Place the boat containing the sample on the shelf directly in front of the closed combustion tube door or into the appropriate position in the autoloader.
    d. For manual loading systems, initiate the analysis sequence. When prompted by the software, press the Analyze button and load the combustion boat into the combustion tube until it reaches the sample stop.
    e. For manual loading systems, when the analysis is complete, remove the boat and close the combustion tube door.
    f. For manual loading systems, perform steps 3a through 3e a minimum of three times for each reference material used.
    g. For auto-loading systems, perform steps 3a through 3c a minimum of three times for each reference material used and initiate the analysis sequence.
    h. Calibrate the instrument following the procedure outlined in the operator's instruction manual.
    i. Verify the calibration by analyzing 0.10 to 0.25 g of a reference material, different from the reference material used for calibration, following steps 3a through 3e. Confirm that the results are within the acceptable tolerance range.
4. Determine the sample blank.
    a. From the Analysis Screen, use the Login Bar to add three or more blank replicates.
    b. Select "TOC Analysis" as the Method and "TOC, ROC, and TIC Determination" as the Furnace Step Method (parameters noted above).
    c. Analyze blank replicates following steps 2c through 2g a minimum of three times.
    d. Set the blank for the samples following the procedure outlined in the operator's instruction manual.
5. Analyze the samples.
    a. Weigh 0.15 to 0.25 g of sample into a pre-baked combustion boat. Enter sample mass and identification into the Analysis Screen.
    b. Select "TOC Analysis" as the Method and "TOC, ROC, and TIC Determination" as the Furnace Step Method (parameters noted above).
    c. Place the boat containing the sample on the shelf directly in front of the closed combustion tube door or into the appropriate position in the autoloader.
    d. For manual loading systems, initiate the analysis sequence. When prompted by the software, press the Analyze button and load the combustion boat into the combustion tube until it reaches the sample stop.
    e. For manual loading systems, when analysis is complete, remove the boat and close the combustion tube door.
    f. For manual loading systems, perform steps 5a through 5e for each sample to be analyzed.
    g. For auto-loading systems, perform steps 5a through 5c for each sample to be analyzed and initiate the analysis sequence.

Typical Data

The low carbon calibration was performed utilizing a linear, force through origin calibration using LECO 502-696 (Lot 1005) Synthetic Carbon LCRM (1.01 % C). The high carbon calibration was performed utilizing a linear, full regression calibration using LECO 502-696 (Lot 1005) Synthetic Carbon LCRM (1.01 % C) and LECO 502-905 (Lot 1002) Synthetic Carbon LCRM (5.00 % C). The calibrations were verified using LECO 502-934 (Lot 1002) Synthetic Carbon LCRM (0.53 % C) and a reduced sample mass of LECO 502-905 (Lot 1002) Synthetic Carbon LCRM (5.00 % C). The results are reported on a dry basis.

Sample	*Mass (g)*	*% TC*	*% TOC*	*% ROC*	*% TIC*	*% TOC + ROC*
NAPT Soil Sample 1‡	0.2526	4.12	2.34	0.76	1.02	3.10
2019-116	0.2528	4.23	2.44	0.77	1.01	3.21
3.31 % ± 0.346 % TOC‡‡	0.2496	4.09	2.30	0.78	1.01	3.08
	0.2512	4.02	2.34	0.71	0.96	3.05
	0.2494	4.22	2.45	0.78	0.99	3.23
	$\bar{x}$ =	***4.13***	***2.38***	***0.76***	***1.00***	***3.14***
	s =	***0.09***	***0.07***	***0.03***	***0.02***	***0.08***
NAPT Soil Sample 2‡	0.2496	1.20	0.97	0.20	0.02	1.17
2021-113	0.2510	1.21	0.98	0.21	0.02	1.19
1.13 % ± 0.030 % TOC‡‡	0.2485	1.17	0.96	0.18	0.02	1.14
	0.2530	1.20	0.99	0.19	0.02	1.18
	0.2502	1.16	0.96	0.18	0.02	1.14
	$\bar{x}$ =	***1.19***	***0.97***	***0.19***	***0.02***	***1.16***
	s =	***0.02***	***0.01***	***0.01***	***0.01***	***0.02***
NAPT Soil Sample 3‡	0.2505	4.23	2.48	0.78	0.97	3.26
2022-104	0.2523	4.19	2.40	0.77	1.03	3.17
3.08 % ± 0.215 % TOC‡‡	0.2504	4.21	2.47	0.76	0.98	3.23
	0.2504	4.23	2.44	0.78	1.01	3.22
	0.2516	4.23	2.45	0.76	1.03	3.21
	$\bar{x}$ =	***4.22***	***2.45***	***0.77***	***1.00***	***3.22***
	s =	***0.02***	***0.03***	***0.01***	***0.03***	***0.04***

$\bar{x}$ = Sample Mean; s = Sample Standard Deviation

‡Soil Sample was obtained from the North American Proficiency Testing (NAPT) Program, Soil Science Society of America.

‡‡Value was obtained from the North American Proficiency Testing (NAPT) Program, Soil Science Society of America. This value is the TOC and ROC values combined. It is not a certified value and is an informational value only. The margin of error is a Mean Absolute Deviation (MAD) value.

LECO Corporation | 3000 Lakeview Avenue | St. Joseph, MI 49085 | Phone: 800-292-6141 | 269-985-5496
info@leco.com • www.leco.com | ISO-9001:2015 Certified|

Form No. 203-821-728 8/26—REV0