Temperature-Dependent Determination of Total Organic Carbon (TOC) in Soil, Rock, and Shale

LECO Corporation; Saint Joseph, Michigan USA

Instrument: RC612 Series

Introduction

Total Organic Carbon (TOC) in soil, rock, and shale is a common analysis used for determining locations of natural hydrocarbon fossil fuel deposits, quality control of raw building materials, and assessment of soil quality in agriculture. Direct measurement of TOC is difficult due to the unique properties of soil, rock and shale and must be performed using either an acid digestion or temperature dependent differentiation method. The acid digestion method utilizes an inorganic non-oxidizing acid like hydrochloric acid, which is not strong enough to effect the organic carbon, to digest the carbonates present in the sample to carbon dioxide gas, leaving the TOC intact. The determination is then calculated by difference (TOC = Total Carbon - TIC). The temperature-dependent method requires no sample pretreatment and no additional sample preparation, removing hazardous chemicals and time consuming acid digestions from the process. With the assumption that organic species decompose to CO_2 between 150 °C to 450 °C, and inorganic species below 1000 °C, heating to these specific temperatures in an oxidizing environment allows differentiation of the TOC from the Total Inorganic Carbon (TIC).

The LECO RC612, with its variable ramp furnace, wide range carbon detection range and simultaneous carbon and moisture determination, is optimized for the temperature differentiation analysis of forms of carbon in soil, rock and shale. The following application note outlines the instrument parameters and procedure for temperature dependent carbon determination with the RC612.

Accessories

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

Reference Materials

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

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

Method Parameters: TOC Analysis

Analysis Parameters

Carrier Gas:	Oxygen
Purge Flow:	3.00 lpm
Analysis Flow:	0.75 lpm
Catalyst Heater Temp:	850 °C
Afterburner Temp:	850 °C

Element Parameters	*Carbon*	*Water*
Analyze:	Yes	No
Conversion Factor:	1.00	1.00
Significant Digits:	5	5

Carbon Range:	Auto
Switch Level to High Cell:	37500
Switch Level to Low Cell:	35000
IR Baseline Time:	2 seconds
Endline Time:	2 seconds

Furnace Step: Synthetic Carbon Calibration

Step	*Name*	*Target (°C)*	*Ramp*	*Hold (s)*	*Est Time (s)*
Start	Synthetic Carbon	1000	N/A	Synthetic Carbon	100 to 600

Hold Parameters: Synthetic Carbon

	Carbon	*High Carbon*
Min Analysis Time:	100	100
Peak Threshold:	0	0
Comparator Level:	0.30	0.30
Max Analysis Time:	600	600

Furnace Steps: TOC Sample Analysis

Step	*Name*	*Target (°C)*	*Ramp*	*Hold (s)*	*Est Time (s)*
Start	Start	105	N/A	0	0
1	Organic	450	120.00	300	648
2	Inorganic	1000	120.00	100	567

Procedure

1. Prepare the instrument as outlined in the operator's instruction manual.
2. Determine Blank.
   Note: 781-335 Quartz Crucibles and 625-505-430 Nickel Crucibles, should be pre-baked at 1100 °C, to remove any residual carbon, and cooled in a desiccator until time of analysis. Handle the crucibles with clean tongs only.
   a. Enter 1.0000 g mass into Sample Login (F3) using Blank as the sample name, select the number of replicates, "TOC Analysis" as the Method, and "Synthetic Carbon Calibration" as the Furnace Step Method (parameters noted above).
   b. Place a "clean" crucible on the shelf directly in front of the closed combustion tube door, or into the appropriate autoloader location, and initiate the analysis sequence (F5).

Inorganic Application Note

   c. When the load sample message appears, select "Ok" in the message box, open the door, load the crucible into the combustion tube until it reaches the sample stop, remove the sample puller, and close the door.
   d. When analysis is complete, remove the crucible and close the combustion tube door.
   e. Repeat steps 2a through 2d a minimum of three times.
   f. Set the blank following the procedure outlined in the operator's instruction manual.
3. Calibration.
   a. Weigh 0.10 to 0.25 g of the selected reference material into a "clean" crucible.
   b. Enter mass and sample identification into Sample Login (F3), select "TOC Analysis" as the Method, and "Synthetic Carbon Calibration" as the Furnace Step Method (parameters noted above).
   c. Place the crucible, containing the sample, on the shelf directly in front of the closed combustion tube door, or into the appropriate autoloader location, and initiate the analysis sequence (F5).
   d. When the load sample message appears, select "Ok" in the message box, open the door, load the crucible into the combustion tube until it reaches the sample stop, remove the sample puller, and close the door.
   e. When analysis is complete, remove the crucible and close the combustion tube door.
   f. Repeat steps 3a through 3e a minimum of three times for each reference material used.
   g. Calibrate the instrument using single standard curve following the procedure outlined in the operator's instruction manual.
   h. Verify the calibration by analyzing 0.10 to 0.25 g of a reference material, different than the material used for calibration, following steps 3a through 3e.
4. Determine Sample Blank.
   a. Enter 1.0000 g mass into Sample Login (F3) using Blank as the sample name, select the number of replicates, "TOC Analysis" as the Method, and "TOC Sample Analysis" as the Furnace Step Method (parameters noted above).
   b. Analyze Blank following steps 2b through 2d a minimum of three times.
   c. Set the blank following the procedure outlined in the operator's instruction manual.
5. Analyze Samples.
   a. Weigh 0.15 to 0.25 g of sample into a "clean" crucible.
   b. Enter mass and sample identification into Sample Login (F3), select "TOC Analysis" as the Method, and "TOC Sample Analysis" as the Furnace Step Method (parameters noted above).
   c. Analyze the sample following steps 3c through 3e outlined above.

Method Equation Parameters

Equation Name	*Equation Formula*
Organic Carbon %	@PeakCO2("Organic")
Inorganic Carbon %	@PeakCO2("Inorganic")

Typical Results

Name	*Mass (g)*	*Total Carbon (%)*	*Organic Carbon (%)*	*Inorganic Carbon (%)*
NIST 886	0.2498	5.59	0.236	5.36
Refractory Gold Ore	0.2503	5.57	0.240	5.33
Total Carbon: 5.7%	0.2481	5.55	0.234	5.32
Inorganic Carbon: 5.4%	0.2504	5.60	0.223	5.38
	0.2502	5.58	0.233	5.34
	Avg =	***5.58***	***0.233***	***5.34***
	s =	***0.02***	***0.006***	***0.02***
502-309 Lot: 1012	0.1564	11.80	11.42	0.384
Soil	0.1559	11.75	11.38	0.375
Total Carbon:	0.1507	11.82	11.44	0.380
11.98 ±0.44%	0.1520	11.76	11.36	0.396
	0.1542	11.60	11.27	0.332
	Avg =	***11.75***	***11.37***	***0.373***
	s =	***0.09***	***0.07***	***0.024***
502-308 Lot: 1018	0.2616	2.39	2.21	0.181
Soil	0.2508	2.41	2.21	0.191
Total Carbon:	0.2541	2.40	2.21	0.188
2.42 ±0.05%	0.2537	2.34	2.14	0.196
	0.2525	2.34	2.14	0.199
	Avg =	***2.37***	***2.18***	***0.191***
	s =	***0.03***	***0.04***	***0.007***
Core Drilling 1	0.1525	15.16	14.82	0.343
	0.1516	14.99	14.66	0.330
	0.1521	15.01	14.67	0.339
	0.1528	15.00	14.67	0.324
	0.1527	15.02	14.69	0.326
	Avg =	***15.04***	***14.70***	***0.332***
	s =	***0.07***	***0.06***	***0.008***
Core Drilling 2	0.1544	11.30	11.09	0.208
	0.1505	11.25	11.03	0.216
	0.1507	11.29	11.06	0.219
	0.1522	11.38	11.17	0.213
	0.1519	11.19	10.98	0.212
	Avg =	***11.28***	***11.07***	***0.213***
	s=	***0.07***	***0.07***	***0.004***

**Results reported on a dry basis. Low carbon calibration was performed utilizing a single standard force through origin calibration using LECO 502-696 1% Synthetic Carbon. High carbon calibration was performed utilizing a linear, full regression calibration using LECO 502-905 5% Synthetic Carbon and LECO 502-902 Calcium Carbonate.*

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