Application Note

Instrument: RC612

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Temperature-Dependent Determination of Total Organic Carbon (TOC) in Geological Materials

LECO Corporation; St. Joseph, Michigan USA

Introduction

Total Organic Carbon (TOC) determination in geological materials—such as metal-bearing ores, oil shales, minerals, and aggregates—is routinely performed to assess organic matter content. This is an essential quality control procedure used to identify locations of natural hydrocarbon fuel deposits and to ensure the quality and compositional integrity of raw materials used in construction.

The organic Carbon content of rock and shale samples is one of the most important parameters used in the evaluation of sediments as a potential source for petroleum or natural gas. TOC content controls the hydrocarbon generation potential of the rock and the content of adsorbed natural gas within the rock, as it is the source material responsible for the formation of natural hydrocarbon fossil fuel deposits.

The determination of TOC is also an important quality control procedure used during the manufacturing of cement, from the raw material to the final product. TOC content is used to monitor the quality of raw materials such as limestone and shale and manage the kiln operation. This allows manufacturers to ensure that the final product meets the requirements necessary for optimal performance and compliance with environmental regulations.

Method Summary

Direct measurement of TOC is difficult due to the unique properties of geological materials. Therefore, analysis must be performed using either an acid digestion method or temperature-dependent differentiation method. The acid digestion method utilizes an inorganic non-oxidizing acid like hydrochloric acid, (which is not strong enough to have an effect on the organic Carbon) leaving the TOC content intact. However, the carbonates present in the sample will be digested into Carbon Dioxide gas.

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 of Carbon combust to form CO_2 between 150 °C to 450 °C, and inorganic species below 1000 °C, heating to these specific temperatures in an oxidizing environment allows for the differentiation of the TOC content from the Total Inorganic Carbon (TIC) content.

Instrument Model and Configuration

The LECO RC612 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 or decompose. This capability facilitates the temperature differentiation determination of the different species of Carbon in geological materials. The following application note outlines the instrument parameters and procedure for temperature-dependent Carbon determination using the RC612.

Accessories

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

Note: 781-335 Quartz Boats and 625-505-430 Nickel Boats should be pre-baked at 1100 °C to remove any residual Carbon and cooled in a desiccator until time of analysis. 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.

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: Synthetic Carbon Calibration 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.

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.
   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 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.
   c. For manual loading systems, initiate the analysis sequence (F5). When the load sample message appears, select "Ok" in the message box, open the door, load the boat into the combustion tube until it reaches the sample stop, remove the sample puller, and close the door.
   d. For manual loading systems, when analysis is complete, remove the boat and close the combustion tube door.
   e. For manual loading systems, perform steps 2a through 2d a minimum of three times.
   f. For auto-loading systems, perform steps 2a through 2b a minimum of three times, and initiate the analysis sequence.
   g. 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 pre-baked boat.
   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 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 (F5). When the load sample message appears, select "Ok" in the message box, open the door, load the boat into the combustion tube until it reaches the sample stop, remove the sample puller, and close the door.
   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 using a single standard curve, 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 material used for calibration, following steps 3a through 3g. Confirm that the results are within the acceptable tolerance range.
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 replicates following steps 2b through 2f 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 pre-baked boat.
   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. 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 (F5). When the load sample message appears, select "OK" in the message box, pen the door, load the boat into the combustion tube until it reaches the sample stop, remove the sample puller, and close the door.
   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.

Method Equation Parameters

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

Typical Results

The low Carbon calibration was performed utilizing a single standard, linear, force through origin calibration, using LECO 502-029 Synthetic Carbon (1.00 % C). The high Carbon calibration was performed utilizing a linear, full regression calibration, using LECO 502-030 Synthetic Carbon (4.99 % C) and LECO 501-034 Calcium Carbonate (12.01 % C). The results are reported on a dry basis.

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
	$\bar{x}$ =	***5.58***	***0.233***	***5.34***
	s =	***0.02***	***0.006***	***0.02***
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
	$\bar{x}$ =	***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
	$\bar{x}$ =	***11.28***	***11.07***	***0.213***
	s=	***0.07***	***0.07***	***0.004***

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

LECO Corporation | 3000 Lakeview Avenue | St. Joseph, MI 49085 | Phone: 800-292-6141 | 269-985-5496
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