Carbon and Sulfur Determination in Limestone/Dolomite

LECO Corporation; Saint Joseph, Michigan USA

Instrument: CS844 Series

Introduction

Limestone and dolomite are sedimentary rocks with an enormous diversity of uses in a variety of fields. Limestone is believed to have more uses than any other single rock known to man, which includes construction material, aggregate, fluxing agent, acid neutralizing agent, and an animal feed filler. When calcined, lime [CaO] is created, which is a more effective acid-neutralization agent and construction raw material. Dolomite is harder than limestone, making it a superior construction material when used for dimensional stone or aggregate. Limestone is composed primarily of calcium carbonate [$CaCO_3$] and is known as the mineral calcite. Dolomite is similar to limestone, and is primarily composed of calcium magnesium carbonate [$CaMg(CO_3)_2$]. Due to the variety of uses for these materials, confirmation of material purity is a key quality control metric. Total carbon and sulfur content are the most common metrics used to confirm purity by the industry due to the simplicity and reliability of the test procedure. The preferred method for total carbon and sulfur determination is combustion in an induction furnace, where sulfur is oxidized to SO_2 and carbon to CO_2. Total carbon and sulfur are calculated relative to the amount of SO_2 and CO_2 produced. With its high efficiency furnace, heated dust filter, dual range detection system, and high precision and accuracy, the CS844 is a smart addition for any industrial laboratory. The following application note outlines the settings and steps required to determine the total carbon and sulfur levels in limestone and dolomite with the CS844.

Sample Preparation

Samples should be crushed to a uniform powder prior to analysis.

Accessories

528-018 or 528-018HP Crucible (preheated*); 763-266 LECOCEL, 501-078 Iron Powder, 501-636-HAZ V_2O_5 Accelerator; 773-579 Metal Scoop; 761-929 Tongs

**For optimal precision, ceramic crucibles must be preheated in a muffle or tube furnace (LECO TF4) at ≥1250 °C for a minimum of 15 minutes or at ≥1000 °C for a minimum of 1 hour. Crucibles must be handled with clean tongs to avoid contamination. The crucibles are removed from the furnace, allowed to cool for 1 to 2 minutes, and then are transferred to a desiccator for storage. Crucibles should be reheated if not used within four hours.*

Calibration

502-902 Calcium Carbonate LCRM®, 502-909 Metal Bearing Ore LCRM; NIST or other suitable Reference materials may be used as well.

Method Parameters

General Parameters

Purge Time:	10 s
Analysis Delay	20 s
Sample Cool Time:	0 s
Furnace Mode:	Constant
Furnace Power:	100%
Furnace Ramp Rate:	0

Element Parameters	*Carbon*	*Sulfur*
Integration Delay:	0 s	0 s
Starting Baseline:	2 s	2 s
Use Comparator:	Yes	Yes
Comparator Level:	5.00%	2.00%
Min Integration Time:	50 s	50 s
MaxIntegration Time:	80 s	80 s
Significant Digits:	5	5

Procedure

1. Prepare the instrument and crucibles as outlined in the operator's instruction manual.
2. Determine the instrument blank.
   a. Login a minimum of 3 Blank reps.
   b. Add ~0.4 g of 501-078 Iron Powder, ~0.6 g 501-636-HAZ V_2O_5 to a 528-018 or 528-018HP Crucible and thoroughly mix.
   c. Add ~3.0 g of LECOCEL to crucible.
   d. Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable), and initiate analysis.
   e. Repeat steps 2b through 2d a minimum of three times.
   f. Set the blank by following the procedure outlined in the operator's instruction manual.
3. Calibrate/Drift Correct
   a. Login a minimum of 3 Standard reps.
   b. Add ~0.4 g of 501-078 Iron Powder, ~0.6 g 501-636-HAZ V_2O_5 to a 528-018 or 528-018HP crucible and thoroughly mix. Tare the crucible and accelerators.
   c. Weigh ~0.1 to ~0.2 g of suitable calibration/drift Reference Material into the crucible and thoroughly mix; enter the mass and standard identification of the standard.
   d. Add ~3.0 g of LECOCEL to crucible.
   e. Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable) and initiate analysis.

Inorganic Application Note

f. Repeat steps 3b and 3e a minimum of three times for each calibration/drift Reference Material intended for calibration/drift.
g. Calibrate/drift correct by following the procedure outlined in the operator's instruction manual.

4. Sample Analysis
   a. Login a Sample with appropriate number of reps.
   b. Add ~0.4 g of 501-078 Iron Powder, ~0.6 g 501-636-HAZ V_2O_5 to a 528-018 Crucible and thoroughly mix. Tare the crucible and accelerators.
   b. Weigh ~0.1 g to 0.15 g Limestone/Dolomite sample into the crucible thoroughly mix; enter the mass and sample identification of the crucible.
   c. Add ~3.0 g of LECOCEL to crucible.
   e. Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable), and initiate analysis.

Typical Results

Sample	*Mass (g)*	*Carbon (%)*	*Sulfur (%)*
Dolomite	0.1494	12.58	0.021
	0.1506	12.58	0.021
	0.1504	12.57	0.021
	0.1500	12.60	0.022
	0.1514	12.58	0.021
	Avg =	***12.58***	***0.021***
	s =	***0.01***	***<0.001***
LCRM 502-902	0.1496	11.99	0.003
$CaCO_3$	0.1503	12.04	0.003
12.01 ± 0.03%	0.1502	11.99	0.003
	0.1501	12.02	0.003
	0.1498	12.01	0.003
	Avg =	***12.01***	***0.003***
	s =	***0.02***	***<0.001***
NIST SRM 1d	0.1480	11.40	0.100
Argillaceous	0.1483	11.40	0.096
Limestone	0.1503	11.38	0.097
11.50% C	0.1505	11.40	0.102
0.1028% S	0.1498	11.41	0.100
	Avg =	***11.40***	***0.099***
	s =	***0.01***	***0.002***

**Calibrated with LCRM 502-902 @ 12.01% Carbon, LCRM 502-909 @ 0.68% Sulfur using linear forced through origin calibrations.*

LECO Corporation | 3000 Lakeview Avenue | St. Joseph, MI 49085 | Phone: 800-292-6141 | 269-985-5496
info@leco.com • www.leco.com | ISO-9001:2008 HQ-Q-994 | *LECO is a registered trademark of LECO Corporation.*
LCRM = LECO Certified Reference Materials and is a registered trademark of LECO Corporation.

