Application Note

Instrument: CS744 Series

Carbon and Sulfur Determination in Limestone/Dolomite

LECO Corporation; Saint Joseph, Michigan USA

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 wide detection range and easy-to-use touch-screen interface, the CS744 sulfur by combustion analyzer makes the perfect addition to 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 CS744.

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 ~1.5 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 ~1.5 g of LECOCEL to crucible.
   e. Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable) and initiate analysis.
   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 ~1.5 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 (%)*
NIST SRM 1c	0.1489	10.98	0.124
Argillaceous	0.1513	10.96	0.118
Limestone	0.1493	10.98	0.119
	0.1495	10.99	0.123
	0.1511	10.97	0.120
	Avg =	***10.98***	***0.121***
	s =	***0.01***	***0.003***
Dolomite	0.1503	12.57	0.020
	0.1504	12.58	0.021
	0.1514	12.58	0.020
	0.1511	12.47	0.020
	0.1532	12.55	0.020
	Avg =	***12.55***	***0.020***
	s =	***0.05***	***<0.001***
LCRM 502-902	0.1494	12.00	0.003
$CaCO_3$	0.1499	11.98	0.003
12.01 ± 0.03%	0.1499	12.02	0.003
	0.1498	12.03	0.003
	0.1505	12.00	0.003
	Avg =	***12.01***	***0.003***
	s =	***0.02***	***<0.001***
NIST SRM 1d	0.1501	11.38	0.105
Argillaceous	0.1495	11.40	0.108
Limestone	0.1507	11.37	0.107
11.50% C	0.1497	11.40	0.106
0.1028% S	0.1517	11.39	0.108
	Avg =	***11.39***	***0.107***
	s =	***0.01***	***0.001***

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

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