Carbon and Sulfur in Lime

LECO Corporation; Saint Joseph, Michigan USA

Instrument: CS744

Introduction

Lime is a common classification of materials representing the manufactured forms of lime, quicklime and hydrated lime. It does not include limestone, the raw material for lime manufacturing. Quicklime is formed from the calcination of limestone and primarily consists of calcium oxide (CaO) with varying amounts of magnesium oxide (MgO). Quicklime is further defined by the ratio of MgO relative to CaO. Hydrated lime* is formed by adding water to quicklime and allowing the oxides to convert to hydrides. Hydrated lime is further classified by the amount of chemically combined water.

**Hydrated lime, like other materials with elevated crystalline moisture content, can not be analyzed by the CS744 or other induction-based techniques.*

The amount of carbon present in lime is used as a means of classifying the degree of calcination, a quality control process parameter. It can also be used to determine the amount of reabsorbed carbon dioxide (CO_2) in the lime. Sulfur is an unwanted contaminant. Subsequently, the amount of sulfur present is used as a measure of lime purity. The CS744 carbon and sulfur elemental analyzer can quickly and simultaneously determine the amount of carbon and sulfur present in all forms of lime. The following application note outlines the sample preparation requirements, common accessories, recommended calibration samples, method parameters, operational procedures, and typical results for the determination of carbon and sulfur in lime with a CS744.

Sample Preparation

Samples should be a uniform powder, preferably passing through a 100 mesh (150 micron) sieve. Some Reference Materials require drying; see the Certificate of Analysis for drying instructions, if applicable.

Accessories

528-018 or 528-018HP Ceramic Crucibles*; 763-266 LECOCEL or 763-263 LECOCEL III accelerator; and 501-077 or 502-231 Iron Chip accelerator, 773-579 Metal Scoop.

**For best precision, ceramic crucibles should be baked in a muffle or tube furnace (LECO TF10) at a minimum of 1250°C for a minimum of 15 minutes, or at 1000°C for 40 minutes. The crucibles are removed from the furnace, allowed to cool for 1 to 2 minutes, and transferred to a desiccator for storage. If the crucibles are not used within four hours, they should be re-baked. After baking, handle crucibles with clean tongs only. Do not use fingers.*

Calibration

LECO 502-319 Ore Tailings, LECO 502-029 Synthetic Carbon, NIST SRM 2690 Fly Ash, or other suitable reference materials.

Method Parameters

Analysis Parameters

Purge Time:	15 s
Analysis Delay:	20 s
Sample Cool Time:	0 s
Furnace Power:	100%

Element Parameters

	Carbon	*Sulfur*
Integration Delay:	0 s	0 s
Starting Baseline:	2 s	2 s
Use Comparator:	No	No
Integration Time:	50 s	60 s
Use Endline:	Yes	Yes
Ending Baseline:	2 s	2 s

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Determine the instrument Blank.
   a. Login a minimum of three Blank reps.
   b. Add one (773-579) scoop (~1.2 g) of LECOCEL or LECOCEL III accelerator to the crucible.
   c. Add one (773-579) scoop (~0.8 g) of Iron Chip accelerator to the crucible.
   d. Place the crucible on the pedestal or appropriate autoloader position.
   e. Initiate the analysis by pressing the Analyze button.
   f. Repeat steps 2b through 2e a minimum of three times.
   g. Set the Blank according to the procedure outlined in the operator's instruction manual.
3. Instrument calibration/drift correction.
   a. Login a minimum of three Standard reps for each calibration/drift reference material to be used for calibration/drift.
   b. Weigh ~0.2 to 0.25 g of a calibration/drift reference material into the crucible and enter the mass and reference material identification into the standard login.
   c. Add one (773-579) scoop (~1.2 g) of LECOCEL or LECOCEL III accelerator on top of the reference material.
   d. Add one (773-579) scoop (~0.8 g) of Iron Chip accelerator on top of the reference material.
   e. Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable), and initiate analysis.

Inorganic Application Note

f. Repeat steps 3b through 3e a minimum of three times for each calibration/drift standard intended for calibration/drift.
g. Calibrate/drift correct by following the procedure outlined in the operators instruction manual.

4. Analyze Samples.
   a. Login a Sample with the desired number of reps.
   b. Weigh ~0.2 to 0.25 g of sample into the crucible and enter the mass and sample identification into the sample login.
   c. Add one (773-579) scoop (~1.2 g) of LECOCEL or LECOCEL III accelerator on top of the sample.
   d. Add one (773-579) scoop (~0.8 g) of Iron Chip accelerator on top of the sample.
   e. Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable), and initiate analysis.
   f. Repeat steps 4a through 4e as necessary.

Typical Results

Description	*Mass (g)*	*% Carbon*	*% Sulfur*
502-319	0.2153	1.53	1.11
1.53% Carbon	0.2194	1.52	1.09
1.10% Sulfur	0.2322	1.55	1.08
Ore Tailings	0.2308	1.52	1.12
	0.2428	1.54	1.10
	$\bar{\chi}$=	***1.53***	***1.10***
	***s*=**	***0.01***	***0.02***
High	0.2009	1.41	0.021
Calcium Lime	0.2259	1.40	0.025
	0.2125	1.40	0.026
	0.2344	1.42	0.027
	0.2101	1.41	0.027
	$\bar{\chi}$=	***1.41***	***0.025***
	***s*=**	***0.01***	***0.002***
Dolomitic Lime	0.2037	0.303	0.048
	0.2059	0.305	0.043
	0.2313	0.307	0.044
	0.2259	0.313	0.042
	0.2152	0.312	0.043
	$\bar{\chi}$=	***0.308***	***0.044***
	***s*=**	***0.004***	***0.002***

Carbon Detector calibrated with 502-319 using a single standard force through origin calibration.
Sulfur Detector calibrated with 502-319 using a single standard force through origin calibration.

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