Determination of Carbon and Sulfur in Fluorspar

LECO Corporation; Saint Joseph, Michigan USA

Instrument: CS844 Series

Introduction

Fluorspar, a mineral composed of calcium fluoride (CaF_2), is an important material in many industrial processes. It is used as a fluxing agent for steelmaking, a raw material for making aluminum fluoride and hydrofluoric acid, and in the production of enamels, glasses, and cement. As with any pure or graded processing material, the purity of the material must be tightly characterized in order to control the downstream effects of the impurities on the final product. Carbon is one of the impurities in fluorspar that must be determined, and can be in the form of carbonates or organic carbon. The LECO CS844, when equipped with the optional halogen trap, provides a fast and accurate means of determining the amount of total carbon in fluorspar, and can be set to automatically calculate the approximate carbon as $CaCO_3$ in the sample. The following application note outlines the sample preparation steps, analytical procedure, and provides examples of typical results from the CS844 on known certified reference materials.

Sample Preparation

Surface contamination on the sample can cause significant errors in the analytical data; therefore, care must be taken to ensure a clean, representative sample is analyzed. Solid samples should be crushed to a uniform mesh size prior to analysis.

Accessories

528-018 or 528-018HP Crucible (baked*); 502-173 LECOCEL II HP; 502-231 Iron Chip Accelerator; 775-579 Metal Scoop; 761-929 Tongs; 619-592-150 Chlorine/Fluorine Trap Kit

**Ceramic crucibles are baked in a muffle or tube furnace (LECO TF-10) at 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 are then transferred to a desiccator for storage. If the crucibles are not used within four hours, they should be re-baked.*

Calibration

There are several suitable LRM® and LCRM® steel reference materials available from LECO (501-933, 501-935, 502-402, 502-809). Likewise, NIST, JK, JSS, and BCS are certified bodies that have a variety of certified reference materials (SRM/CRM) available as well. It is important that both high- and low-carbon ranges are calibrated. Linear calibration curves force through origin are recommended. Refer to the operator's instruction manual for details.

NOTE: The oxides produced during combustion of CaF_2 may cause a flow restriction with the autocleaner, therefore it may be necessary to use the MANUAL CLEANER. The downstream pressure on the furnace will decrease if there is a restriction from these oxides.

Method Parameters

General Parameters

Purge Time	15 s
Analysis Delay	30 s
Sample Cool Time	0 s
Furnace Mode	Constant
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	55 s
Use End line	Yes	Yes
Ending Baseline	2 s	2 s
Range Select	Auto	Auto
Range Low	800	800
Range High	950	1200

Procedure

1. Install 619-592-150 Halogen Trap Bracket according to the 619-592-149 Kit Instructions.
   a. Pack the reagent tube for fluorine removal as shown on pages 6-7 of the kit instructions.
2. Prepare the instrument and crucibles as outlined in the operator's instruction manual.
3. Determine the instrument blank.
   a. Login a minimum of three Blank replicates.
   b. Add ~1 g of Lecocel II HP and ~1 g Iron Chip accelerator to the crucible.
   c. Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable), and initiate analysis.
   d. Repeat steps 3b through 3c a minimum of three times.
   e. Set the blank by following the procedure outlined in the operator's instruction manual.
4. Calibrate/Drift Correct
   a. Login a minimum of three Standard replicates.
   b. Weigh ~0.5 g of a steel chip/granular powder calibration/drift reference material into the crucible, and enter the mass and reference material identification of the standard.
   c. Add ~1 g of Lecocel II HP and ~1 g Iron Chip accelerator on top of the reference material.
   d. Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable) and initiate analysis.

Inorganic Application Note

Delivering the Right Results

e. Repeat steps 4b and 4e a minimum of three times for each calibration/drift reference material intended for calibration/drift.
f. Calibrate/drift correct by following the procedure outlined in the operator's instruction manual.

5. Sample Analysis
   a. Login a Sample with appropriate number of replicates.
   b. Weigh ~0.1 to ~0.15 g fluorspar sample into the crucible and enter the mass and sample identification of the sample.
   c. Add ~1 g of Lecocel II HP and ~1 g 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 5b through 5e for all sample replicates.

Typical Results

Sample	*Mass (g)*	*% Carbon*	*% Sulfur*
Fluorspar	0.1511	0.247	0.017
NIST SRM	0.1498	0.236	0.019
79a	0.1503	0.244	0.017
	0.1503	0.245	0.016
	0.1501	0.247	0.018
	0.1506	0.243	0.020
	0.1497	0.234	0.017
	0.1493	0.242	0.017
	0.1491	0.244	0.015
	0.1502	0.242	0.016
	Avg. =	***0.242***	***0.017***
	s =	***0.004***	***0.001***
Fluorspar	0.1494	0.115	0.080
NIST SRM	0.1504	0.115	0.078
180	0.1492	0.114	0.078
	0.1500	0.116	0.079
	0.1500	0.117	0.079
	0.1501	0.114	0.079
	0.1495	0.113	0.078
	0.1501	0.114	0.078
	0.1492	0.115	0.079
	0.1509	0.115	0.078
	Avg. =	***0.115***	***0.079***
	s =	***0.001***	***0.001***

**Calibrated with JK Nr 21 @ 0.1741% Carbon, JK Nr 8F @ 0.0183% Sulfur Steel Chip Certified Reference Material using linear forced through origin calibration.*

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