Application Note

Instrument: 844 Series (C844, CS844, CS844ES)

Determination of Carbon in Silicon Carbide

LECO Corporation; Saint Joseph, Michigan USA

Introduction

Silicon carbide (SiC) is an extremely hard and tough material with numerous industrial applications, ranging from semiconductors and LEDs, to automotive brakes and clutches. The most common production method of SiC involves a carbothermic reduction reaction of silicon dioxide (SiO_2). The determination of total carbon in SiC is an important quality control parameter for all manufacturers. Carbon results are used not only to determine the materials purity, but also to calculate the ratio of SiC and free carbon following the carbothermic reduction reaction.

Instrument Model and Configuration

The 844 instrument series is designed for carbon determination in a wide range of sample materials A pre-weighed sample is combusted in a stream of purified oxygen, using induction to heat the sample. Carbon present in the sample is oxidized to carbon dioxide (CO_2) and swept by the oxygen carrier gas through a heated dust filter, and a drying reagent. The gas flow continues past a heated catalyst, where carbon monoxide (CO) is converted to CO_2, and carbon is then detected as CO_2 by a pair of non-dispersive infrared (NDIR) cells.

Sample Preparation

Samples should be a uniform, representative, powder or granular material.

Accessories

528-018 or 528-018HP Ceramic Crucibles*, 501-263 or 502-492 HP Copper Accelerator, 501-077 or 502-231 HP Iron Chip Accelerator, 773-579 Metal Scoop, and 761-929 Tongs.

**For optimal precision, ceramic crucibles should be heated in a muffle or tube furnace (such as a LECO TF4) at 1350 °C for a minimum of 20 minutes. The crucibles should be removed from the furnace, allowed to cool for one to two minutes, and then transferred to a desiccator for storage until use. Crucibles should be reheated if not used within four hours. After baking, handle ceramic crucibles with clean tongs only; do not use fingers.*

Reference Materials

LCRM®, LRM®, NIST, or other suitable reference materials.

*Method Parameters***

Parameter	
Purge Time	10 s
Delay Time	20 s
Sample Cool Time	10 s
Furnace Power	100%
Nominal Mass	1.0000 g

*Element Parameters***

Parameter	*Carbon*
Integration Delay	0 s
Starting Baseline	2 s
Use Comparator	No
Integration Time	60 s
Use Endline	Yes
Ending Baseline	2 s
Range Select	Auto
Range Lower Limit	800
Range Upper Limit	950

***Refer to 844 Series Operator's Instruction Manual for Parameter definitions.*

Procedure

1. Prepare the instrument for operation as outlined in the operator's instruction manual.
2. Determine Blank.
   a. Log in a minimum of three Blank replicates.
   b. Add ~1.0 g of 501-263 or 502-492 HP Copper Accelerator and ~1.5 g of 501-077 or 502-231 HP Iron Chip Accelerator to a preheated 528-018 or 528-018HP Ceramic Crucible.
   c. Place the crucible on the furnace pedestal, or in the appropriate autoloader position (if applicable) and initiate the analysis sequence.
   d. Perform steps 2b through 2c a minimum of three times.
   e. Set the blank following the procedure outlined in the Operator's Instruction Manual.
3. Calibrate/Drift Correct.
   a. Log in a minimum of three standard/drift replicates.
   b. Weigh ~0.075 g of a suitable reference material into a preheated ceramic crucible.
   c. Enter the reference material mass and identification into the Login screen.
   d. Tare the ceramic crucible and reference material and add ~1.0 g of copper accelerator and ~1.5 g iron chip accelerator to the crucible, covering the reference material.
   e. Place the crucible on the furnace pedestal, or in the appropriate autoloader position (if applicable) and initiate the analysis sequence.
   f. Perform steps 3b through 3e a minimum of three times for each reference material utilized.
   g. Calibrate/Drift Correct following the procedure outlined in the operator's instruction manual.
   h. Verify the calibration/drift correction by analyzing several replicates of an appropriate mass (~0.075 g) of another/different suitable reference material, following steps 3b through 3e, and confirm that the results are within the acceptable tolerance range.

4. Sample Analysis.
   a. Log in a sample with the desired number of replicates.
   b. Weigh ~0.075 g of sample into a preheated ceramic crucible.
   c. Enter the sample mass and identification into the Login screen.
   d. Tare the ceramic crucible and sample and add ~1.0 g of copper accelerator and ~1.5 g iron chip accelerator to the crucible, covering the sample.
   e. Place the crucible on the furnace pedestal or in the appropriate autoloader position (if applicable) and initiate the analysis sequence.
   f. Perform steps 4b through 4e for each sample replicate being analyzed.

TYPICAL RESULTS

Data was generated utilizing a linear, force through origin calibration using NMIJ 8002-a Silicon Carbide (29.93% C).

Sample	*Mass (g)*	*Carbon (%)*
NMIJ 8001-a	0.0752	29.75
Fine Silicon Carbide Powder	0.0749	29.57
29.80% ± 0.15% C	0.0754	29.70
	0.0754	29.70
	0.0750	29.78
	Avg =	*29.70*
	s =	*0.08*
Reagent Grade Silicon Carbide	0.0755	28.71
99% metals basis	0.0750	28.81
	0.0756	28.82
	0.0752	28.71
	0.0755	28.90
	Avg =	*28.79*
	s =	*0.08*

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