Application Note

Instrument: CS744 Series

Determination of Carbon and Sulfur 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 material's purity, but also to calculate the ratio of SiC and free carbon following the carbothermic reduction reaction. The LECO CS744 series carbon and sulfur determinator is well suited for the simultaneous determination of carbon and sulfur in silicon carbide, combining high precision for carbon and low-level detection for sulfur.

Sample Preparation

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

Accessories

528-018 or 528-018HP Ceramic Crucibles*, 501-263 or 502-492HP Copper Accelerator, 501-077 or 502-231HP Iron Chip Accelerator, 763-266 LECOCEL, 501-078 Iron Powder Accelerator, 501-636-HAZ Vanadium Pentoxide 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*	*Sulfur*
Integration Delay	0 s	0 s
Starting Baseline	2 s	2 s
Use Comparator	No	No
Integration Time	60 s	60 s
Use Endline	Yes	Yes
Ending Baseline	2 s	2 s

***Refer to CS744 Operator's Instruction Manual for Parameter definitions.*

Procedure for Carbon-Only Determination

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 502-492HP Copper Accelerator and ~1.0 g of 501-077 or 502-231HP 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 ~0.1 g of a suitable reference material into a preheated ceramic crucible.
   c. Enter the reference material mass and identification into the Login screen.
   d. Add ~1.0 g of copper accelerator and ~1.0 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 an appropriate mass (~0.1 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 ~0.1 g of sample into a preheated ceramic crucible.
   c. Enter the sample mass and identification into the Login screen.
   d. Add ~1.0 g of copper accelerator and ~1.0 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 for Carbon-Only Determination

Data was generated utilizing a linear, force through origin calibration using Aldrich Lot: MKBJ8589V Silicon Carbide (29.95% C). Samples were analyzed using 502-492HP Copper Accelerator, 502-231HP Iron Chip Accelerator and 528-018HP Ceramic Crucibles.

Sample Name	*Mass (g)*	*Carbon (%)*
Reagent Grade Silicon Carbide	0.1012	29.38
99% Metals Basis	0.1054	29.37
	0.1057	29.31
	0.1034	29.47
	0.1055	29.40
	Avg =	***29.38***
	s =	***0.06***
Silicon Carbide	0.1079	29.82
	0.1033	29.71
	0.1019	29.74
	0.1026	29.83
	0.1015	29.78
	Avg =	***29.77***
	s =	***0.05***

Procedure for Sulfur-Only or Simultaneous Carbon and Sulfur Determination

1. Prepare the instrument for operation as outlined in the operator's instruction manual.
2. Determine Blank.
   a. Login a minimum of three Blank replicates.
   b. Add ~0.4 g of 501-078 Iron Powder Accelerator and ~0.6 g 501-636-HAZ Vanadium Pentoxide Accelerator to a preheated 528-018 or 528-018HP Ceramic Crucible and thoroughly mix.
   c. Add ~1.5 g of 763-266 LECOCEL to the crucible, covering the accelerators.
   d. Place the crucible on the furnace pedestal or in the appropriate autoloader position (if applicable) and initiate the analysis sequence.
   e. Perform steps 2b through 2d a minimum of three times.
   f. 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. Add ~0.4 g of iron powder accelerator and ~0.6 g vanadium pentoxide accelerator to a preheated ceramic crucible. Tare the crucible and accelerators.
   c. Weigh ~0.1 to 0.5 g of a suitable reference material into the crucible and thoroughly mix the reference material with the accelerators.
   d. Enter the reference material mass and identification into the Login screen.
   e. Add ~1.5 g of LECOCEL to the crucible, covering the reference material and accelerators.
   f. Place the crucible on the furnace pedestal or in the appropriate autoloader position (if applicable) and initiate the analysis sequence.
   g. Perform steps 3b through 3f a minimum of three times for each reference material utilized.
   h. Calibrate/Drift Correct following the procedure outlined in the operator's instruction manual.
   i. Verify the calibration/drift correction by analyzing an appropriate mass (~0.1 g) of another/different suitable reference material, following steps 3b through 3f, 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. Add ~0.4 g of iron powder accelerator and ~0.6 g vanadium pentoxide accelerator to a preheated ceramic crucible. Tare the crucible and accelerators.
   c. Weigh ~0.1 g of sample into the crucible and thoroughly mix the sample with the accelerators.
   d. Enter the sample mass and identification into the Login screen.
   e. Add ~1.5 g of LECOCEL to the crucible, covering the sample and accelerators.
   f. Place the crucible on the furnace pedestal or in the appropriate autoloader position (if applicable) and initiate the analysis sequence.
   g. Perform steps 4b through 4f for each sample replicate being analyzed.

Typical Results for Sulfur-Only or Simultaneous Carbon and Sulfur Determination

Data was generated utilizing a linear, force through origin calibration for carbon determination using Aldrich Lot: MKBJ8589V Silicon Carbide (29.95% C). A linear, force through origin calibration was utilized for sulfur determination using LECO 502-904 Lot: 0566 Steel LCRM (0.0179% S). Samples were analyzed using 501-078 Iron Powder Accelerator, 501-636-HAZ Vanadium Pentoxide Accelerator, 763-266 LECOCEL, and 528-018HP Ceramic Crucibles.

Sample Name	*Mass (g)*	*Carbon (%)*	*Sulfur (%)*
Reagent Grade Silicon Carbide	0.1023	29.11	0.0054
99% Metals Basis	0.1025	29.47	0.0057
	0.1014	29.16	0.0053
	0.1040	29.40	0.0052
	0.1036	29.15	0.0050
	Avg =	***29.26***	***0.0053***
	s =	***0.16***	***0.0002***
Silicon Carbide	0.1000	29.65	0.0095
	0.1007	29.54	0.0102
	0.1002	29.56	0.0105
	0.1009	29.73	0.0112
	0.1008	29.53	0.0096
	Avg =	***29.61***	***0.0102***
	s =	***0.09***	***0.0007***

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