Application Note

Instrument: CS844

Carbon and Sulfur Determination in High Carbon Ferroalloys

LECO Corporation; Saint Joseph, Michigan USA

Introduction

Ferroalloys are alloys of iron that contain a high level of one or more other primary elements. The principle ferroalloys consist of silicon, manganese, and chromium that are used as vehicles to introduce the alloying element into the molten metal when making steel or cast iron. For example, silicon is used to deoxidize steel and as an alloying element in cast iron. Manganese is used as an alloying element and mitigates the harmful effects of sulfur in cast iron and steel. Chromium increases corrosion resistance in stainless steels. Since carbon is the most important alloying constituent in steel and cast iron production, and sulfur is a harmful contaminant that negatively affects the mechanical properties of steel and cast iron, the determination of carbon and sulfur levels in the ferroalloy feed stock is a critical quality control parameter.

Sample Preparation

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

Accessories

528-018 or 528-018HP Crucible (previously heated*); 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 are heated in a muffle or tube furnace (such as a LECO TF-4) at 1350° C for a minimum of 20 minutes, or at 1000° C for 40 minutes. 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. After baking, crucibles should only be handled using clean tongs.*

Reference Materials

LCRM®, LRM®, NIST, or other suitable reference materials, such as ferroalloy and steel reference materials.

Method Description

The method utilizes iron powder, vanadium pentoxide, and LECOCEL as accelerators to facilitate combustion. This accelerator combination works well for ferroalloys and may improve sulfur recovery and precision. Even though the carbon blank for this method is considered high, the blank is consistent enough to be properly subtracted from the analysis results. Vanadium pentoxide is considered a hazardous material, therefore, proper precautions should be taken.

Method Parameters

Analysis Parameters

Purge Time	10 s
Analysis Delay	20 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	Yes	Yes
Comparator Level	0.30%	0.30%
Minimum Integration Time	50 s	50 s
Maximum Integration Time	80 s	80 s

Procedure

1. Prepare the instrument as outlined in the operator's instruction manual.
2. Determine the Instrument Blank.
   a. Log in a minimum of three Blank replicates.
   b. Add ~0.4 g of 501-078 Iron Powder and ~0.6 g of 501-636-HAZ V_2O_5 to a previously heated 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 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. Log in a minimum of three Standard replicates.
   b. Add ~0.4 g of 501-078 Iron Powder and ~0.6 g of 501-636-HAZ V_2O_5 to a previously heated crucible. Tare the crucible and accelerators.
   c. Weigh ~0.25 g of an appropriate calibration/drift reference material into the crucible and thoroughly mix.
   d. Enter the mass and sample identification into the appropriate replicate fields.
   e. Add ~1.5 g of 763-266 LECOCEL to the crucible, covering the reference material and accelerators.
   f. Place the crucible on the furnace pedestal (or appropriate autoloader position, if applicable), and initiate analysis.
   g. Repeat steps 3b through 3f a minimum of three times for each calibration/drift reference material being analyzed.
   h. Calibrate/drift correct by following the procedure outlined in the operator's instruction manual.
   i. Verify the calibration by analyzing ~0.25 g of another suitable reference material following steps 3b through 3f and confirm that the results are within an acceptable tolerance.

4. Sample Analysis.
   a. Log in a Sample with an appropriate number of replicates.
   b. Add ~0.4 g of 501-078 Iron Powder and ~0.6 g of 501-636-HAZ V_2O_5 to a previously heated crucible. Tare the crucible and accelerators.
   b. Weigh ~0.25 g of a ferroalloy sample into the crucible and thoroughly mix.
   c. Enter the mass and sample identification into the appropriate replicate fields.
   d. Add ~1.5 g of 763-266 LECOCEL to the crucible, covering the sample and accelerators.
   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***

Sample	*Mass (g)*	*% Carbon*	*% Sulfur*
Ferromanganese	0.2522	6.873	0.0016
Powder	0.2540	6.899	0.0014
	0.2507	6.892	0.0012
	0.2521	6.903	0.0013
	0.2517	6.894	0.0013
	Avg =	***6.892***	***0.0014***
	s =	***0.012***	***0.0001***
Ferrochromium	0.2523	4.138	0.0665
Powder	0.2582	4.170	0.0684
	0.2596	4.173	0.0681
	0.2522	4.245	0.0672
	0.2535	4.224	0.0673
	Avg =	***4.190***	***0.0675***
	s =	***0.043***	***0.0008***

***Results based upon linear, forced through origin calibrations utilizing BCS 208/2 High Carbon Ferro Manganese (6.59% C) and NIST 64c High Carbon Ferro-Chromium (0.0673% S).*

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