Carbon and Sulfur Determination in High Carbon Ferroalloys

LECO Corporation; Saint Joseph, Michigan USA

Instrument: CS230/CS600

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 and are used as vehicles to get 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.

Sampling and Sample Preparation

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

Accessories

528-018 Ceramic Crucibles (preheated)*; 619-880 Crucible Covers (preheated)*; LECOCEL (763-266); Iron Powder (501-078); and V_2O_5 (501-636 HAZ) Accelerator.

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

Method Description

The method utilizes Iron Powder, Vanadium Pentoxide, and LECOCEL as accelerators to facilitate combustion. This method works well for High Carbon Ferroalloys when Sulfur analysis is important. Even though the carbon blank for this method is considered high, the blank is consistent enough to be properly removed from the analysis results. Vanadium Pentoxide is considered a Hazardous Material.

Calibration

NIST, BCS, IARM, LECO, or other suitable reference materials.

Method Parameters

Purge Time (seconds)	10
Delay Time (seconds)	25

Carbon

Minimum Timeout (seconds)	50
Comparator Level	1.00
Significant Digits	5

Sulfur

Minimum Timeout (seconds)	55
Comparator Level	1.00
Significant Digits	5

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Determine blank.
   a. Enter 1.0000 g mass into Sample Login (F3) using Blank as the sample name.
   b. Add ~0.4 g of 501-078 Iron Powder, ~0.6 g 501-636-HAZ V_2O_5 to a preheated 528-018 Crucible and mix thoroughly.
   c. Add ~1.5 g of LECOCEL to crucible.
   d. Place a preheated 619-880 Ceramic Crucible Cover on the 528-018 Crucible.
   e. Place the crucible on the furnace pedestal (or appropriate autoloader position if so equipped) and initiate analysis (F5).
   f. Repeat steps 2a through 2e three to five times.
   g. Enter blank following procedure outlined in operator's instruction manual.
3. Calibrate/Drift.
   a. Add ~0.4 g of 501-078 Iron Powder, ~0.6 g 501-636-HAZ V_2O_5 to crucible and mix thoroughly. Tare the crucible and accelerators.
   b. Weigh ~0.25 g of a suitable reference material into a preheated 528-018 Crucible and mix thoroughly; enter mass and sample identification into Sample Login (F3).
   c. Add ~1.5 g of LECOCEL accelerator to crucible (taking care to evenly cover sample).
   d. Place a preheated 619-880 Ceramic Crucible Cover on the 528-018 Crucible.
   e. Place the crucible on the furnace pedestal (or appropriate autoloader position if so equipped) and initiate analysis (F5).
   f. Repeat steps 3a through 3e three to five times for each calibration/drift sample intended for calibration/drift.
   g. Calibrate/drift using the procedure outlined in the operator's instruction manual.
4. Analyze Samples.
   a. Add ~0.4 g of 501-078 Iron Powder, ~0.6 g 501-636-HAZ V_2O_5 to crucible and mix thoroughly.
   b. Weigh ~0.25 g ferroalloy sample into preheated crucible and mix thoroughly; enter mass and sample identification into Sample Login (F3).
   c. Follow steps 3c through 3e.

Inorganic Application Note

Delivering the Right Results

Typical Results

	Mass	*Carbon %*	*Sulfur %*
NIST 64c	~0.25g	4.706	0.0659
4.68% C		4.691	0.0672
0.0670% S		4.680	0.0661
Ferrochromium		4.683	0.0667
		4.687	0.0677
		4.678	0.0669
		4.697	0.0663
		4.681	0.0676
		4.687	0.0675
		4.693	0.0680
	Average:	***4.688***	***0.0670***
	Std Dev:	***0.009***	***0.0007***
BCS 204/4	~0.25g	5.614	0.0102
5.62% C		5.622	0.0103
0.010% S		5.614	0.0103
Ferrochromium		5.616	0.0102
		5.625	0.0102
		5.630	0.0102
		5.622	0.0100
		5.620	0.0104
		5.624	0.0102
		5.615	0.0102
	Average:	***5.620***	***0.0102***
	Std Dev:	***0.005***	***0.0001***

Calibration: NIST 64c for Sulfur and BCS 204/4 for Carbon with a single standard calibration curve.

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