Carbon and Sulfur Determination in Low 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 II HP (502-173) and Iron Chip HP (502-231) Accelerator or LECOCEL (763-266), Iron Powder (501-078) and V_2O_5 (501-636-HAZ) Accelerator; Metal Accelerator Scoop (773-579).

**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 and covers are not used within four hours, they should be re-baked.*

***Multiple methods are available for the analysis of ferroalloys. Method 1 utilizes LECOCEL II HP and Iron Chip HP Accelerators. Method 2 utilizes LECOCEL, Iron Powder, V_2O_5, and 619-880 Crucible Covers. See Method Selection.*

Method Selection

Multiple methods can be used for the analysis of Carbon and Sulfur in Ferroalloy material on the CS230 or CS600. Method 1 utilizes LECOCEL II and Iron Chip as accelerators.

Method 2 utilizes Iron powder, Vanadium Pentoxide and LECOCEL as accelerators to facilitate combustion. Method 2 works well for Ferroalloys to achieve better Sulfur recovery and precision. 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 for Method 1

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 one 773-579 Metal Scoop of LECOCEL II HP and one scoop of Iron Chip HP accelerator to preheated crucible.
   c. Place the crucible on the furnace pedestal (or appropriate autoloader position if so equipped) and initiate analysis (F5).
   d. Repeat steps 2a through 2c three to five times.
   e. Enter blank following procedure outlined in operator's instruction manual.
3. Calibrate/Drift.
   a. Weigh ~0.25 g of a suitable reference material into preheated 528-018 Crucible; enter mass and sample identification into Sample Login (F3).
   b. Add one 773-579 Metal Scoop of LECOCEL II HP and one scoop of Iron Chip HP accelerator to crucible.
   c. Place the crucible on the furnace pedestal (or appropriate autoloader position if so equipped) and initiate analysis (F5).
   d. Repeat steps 3a through 3c three to five times for each calibration/drift sample intended for calibration/drift.
   e. Calibrate/drift using the procedure outlined in the operator's instruction manual.
4. Analyze Samples.
   a. Weigh ~0.25 g ferroalloy sample into preheated 528-018 Crucible; enter mass and sample identification into Sample Login (F3).
   b. Follow steps 3b through 3c.

Inorganic Application Note

Delivering the Right Results

Procedure for Method 2

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 the 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 V2O5 to crucible and mix thoroughly. Tare the crucible and accelerators.
   b. Weigh ~0.25 g of a suitable reference material into preheated 528-018 Crucible and mix thoroughly; enter mass and sample identification into Sample Login (F3).
   c. Add ~1.5 g of LECOCEL 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 V2O5 to crucible and mix thoroughly. Tare the crucible and accelerators.
   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.

Typical Results

Method1	*Mass*	*Carbon %*	*Sulfur %*
Euro 578-1	~0.25 g	0.015	0.064
0.016% C		0.016	0.065
0.065% S		0.015	0.065
Ferro-		0.016	0.062
Molybdenum		0.016	0.066
		0.014	0.064
		0.015	0.065
		0.015	0.068
		0.016	0.066
		0.016	0.065
	Avg:	***0.016***	***0.065***
	Std Dev:	***0.001***	***0.001***

	Mass	*Carbon %*	*Sulfur %**
NIST 58a	~0.25 g	0.0157	<0.001
0.0143% C		0.0141	<0.001
Ferrosilicon		0.0155	<0.001
		0.0160	<0.001
		0.0140	<0.001
		0.0151	<0.001
		0.0144	<0.001
		0.0169	<0.001
		0.0183	<0.001
		0.0149	<0.001
	Avg:	***0.0155***	***<0.001***
	Std Dev:	***0.0013***	—

		Carbon %	*Sulfur %*
EURO 577-1	~0.25 g	0.087	0.0310
0.089% C		0.090	0.0299
0.034% S		0.088	0.0311
Ferro-		0.088	0.0306
Vanadium		0.089	0.0309
		0.090	0.0292
		0.089	0.0305
		0.089	0.0307
		0.087	0.0288
		0.091	0.0297
	Avg:	***0.089***	***0.0302***
	Std Dev:	***0.001***	***0.0008***

Method 2	*Mass*	*Carbon %*	*Sulfur %*
Euro 578-1	~0.25 g	0.017	0.064
0.016% C		0.017	0.065
0.065% S		0.017	0.065
Ferro-		0.016	0.065
Molybdenum		0.016	0.064
		0.020	0.065
		0.018	0.065
		0.017	0.065
		0.017	0.066
		0.017	0.066
	Avg:	***0.017***	***0.065***
	Std Dev:	***0.001***	***0.001***

	Mass	*Carbon %*	*Sulfur %**
NIST 58a	~0.25 g	0.0151	<0.001
0.0143% C		0.0155	<0.001
Ferrosilicon		0.0158	<0.001
		0.0159	<0.001
		0.0154	<0.001
		0.0167	<0.001
		0.0139	<0.001
		0.0157	<0.001
		0.0153	<0.001
		0.0160	<0.001
	Avg:	***0.0155***	***<0.001***
	Std Dev:	***0.0007***	—

		Carbon %	*Sulfur %*
EURO 577-1	~0.25 g	0.087	0.0308
0.089% C		0.088	0.0310
0.034% S		0.088	0.0308
Ferro-		0.089	0.0309
Vanadium		0.089	0.0312
		0.090	0.0312
		0.089	0.0309
		0.089	0.0314
		0.090	0.0310
		0.088	0.0310
	Avg:	***0.089***	***0.0310***
	Std Dev:	***0.001***	***0.0002***

Calibration: EURO 577-1 for Carbon and Euro 578-1 for Sulfur using a single standard calibration curve.
**Represents results that are below the detection limit for the application.*

LECO Corporation
3000 Lakeview Avenue • St. Joseph, MI 49085 • Phone: 800-292-6141 • Fax: 269-982-8977
info@leco.com • www.leco.com • ISO-9001:2008 HQ-Q-994 • ***LECO is a registered trademark of LECO Corporation.***

