Application Note

Instrument: CS744

Carbon and Sulfur Determination in Low Carbon Ferroalloys

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Introduction

Ferroalloys are alloys of iron that contain a high level of one or more additional 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.

Sample Preparation

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

Accessories

528-018 or 528-018HP Crucibles (previously heated*); 502-173 LECOCEL II, 502-231 Iron Chip Accelerator, 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 Selection

Different methods can be used for the analysis of carbon and sulfur in ferroalloy materials on the CS744. Method 1 utilizes LECOCEL II and iron chip accelerators to facilitate combustion, without the use of hazardous materials.

Method 2 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 removed from the analysis results. Vanadium pentoxide is considered a hazardous material.

Method Parameters

Analysis Parameters		
Purge Time	10 s	
Analysis Delay	20 s	
Sample Cool Time	0 s	
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	1.00%	1.00%
Minimum Integration Time	40 s	40 s
Maximum Integration Time	70 s	70 s

Procedure for Method 1

1. Prepare the instrument as outlined in the operator's instruction manual.
2. Determine the instrument blank.
   a. Login a minimum of three blank replicates.
   b. Add ~1.2 g of 502-173 LECOCEL II and ~0.8 g of 502-231 Iron Chip Accelerator to a previously heated crucible.
   c. Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable), and initiate analysis.
   d. Repeat steps 2b through 2c a minimum of three times.
   e. Set the blank by following the procedure outlined in the operator's instruction manual.
3. Calibrate/Drift Correct.
   a. Login a minimum of three standard replicates for each calibration/drift reference material to be used.
   b. Weigh ~0.25 g of a ferroalloy reference material or ~0.5 g of a steel reference material into a previously heated crucible.
   c. Enter the mass and sample identification into the appropriate replicate fields.
   d. Add ~1.2 g of 502-173 LECOCEL II and ~0.8 g of 502-231 Iron Chip Accelerator on top of the reference material.
   e. Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable) and initiate analysis.
   f. Repeat steps 3b through 3e a minimum of three times for each calibration/drift reference material utilized.
   g. Calibrate/drift correct by following the procedure outlined in the operator's instruction manual.
   h. Verify the calibration by analyzing another suitable reference material following steps 3b through 3e and confirm that the results are within an acceptable tolerance.
4. Sample Analysis.
   a. Login a sample with appropriate number of replicates.
   b. Weigh ~0.25 g of sample into a previously heated crucible.
   c. Enter the mass and sample identification into the appropriate replicate fields.
   d. Add ~1.2 g of 502-173 LECOCEL II and ~0.8 g of 502-231 Iron Chip Accelerator on top of the sample.
   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 for Method 1*

Sample	*Mass (g)*	*% Carbon*	*% Sulfur*
Ferro-Vanadium	0.2489	0.090	0.0301
	0.2495	0.089	0.0300
	0.2492	0.088	0.0289
	0.2481	0.089	0.0303
	0.2508	0.090	0.0297
	***X*=**	***0.089***	***0.0298***
	***s*=**	***0.001***	***0.0005***
NIST 58a	0.2488	0.015	<0.0008
Ferrosilicon	0.2475	0.016	<0.0008
0.0143% ±0.0050% C	0.2501	0.015	<0.0008
	0.2500	0.015	<0.0008
	0.2481	0.017	<0.0008
	***X*=**	***0.016***	***<0.0008***
	***s*=**	***0.001***	**-**
Euro 578-1	0.2486	0.018	0.0639
Ferro-Molybdenum	0.2506	0.017	0.0648
0.016% ±0.002% C	0.2495	0.018	0.0650
0.065% ±0.003% S	0.2483	0.018	0.0645
	0.2499	0.018	0.0647
	***X*=**	***0.018***	***0.0646***
	***s*=**	***0.001***	***0.0004***

**Results based upon linear, forced through origin calibrations utilizing EURO 577-1 Ferro-Vanadium @ 0.089% Carbon and EURO 578-1 Ferro-Molybdenum @ 0.065% Sulfur.*

Procedure for Method 2

1. Prepare the instrument as outlined in the operator's instruction manual.
2. Determine the instrument blank.
   a. Login 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 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. Login 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 and thoroughly mix. Tare the crucible and accelerators.
   c. Weigh ~0.25 g of a ferroalloy reference material or ~0.5 g of a steel reference material into a 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 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 intended for calibration/drift.
   h. Calibrate/drift correct by following the procedure outlined in the operator's instruction manual.
   i. Verify the calibration by analyzing another suitable reference material following steps 3b though 3f and confirm that the results are within an acceptable tolerance.
4. Sample Analysis.
   a. Login a sample with 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 and thoroughly mix. Tare the crucible and accelerators.
   c. Weigh ~0.25 g of a ferroalloy sample 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 crucible, covering the sample and accelerators.
   f. Place the crucible on the furnace pedestal (or appropriate autoloader position if applicable), and initiate analysis.
   g. Repeat steps 4a through 4f as necessary.

Typical Results for Method 2*

Sample	*Mass (g)*	*% Carbon*	*% Sulfur*
Ferro-Vanadium	0.2482	0.090	0.0315
	0.2487	0.089	0.0321
	0.2499	0.088	0.0315
	0.2486	0.086	0.0311
	0.2494	0.086	0.0315
	X=	***0.088***	***0.0315***
	s=	***0.002***	***0.0003***
NIST 58a	0.2487	0.016	<0.0008
Ferrosilicon	0.2485	0.016	<0.0008
0.0143% ±0.0050% C	0.2503	0.016	<0.0008
	0.2479	0.015	<0.0008
	0.2508	0.015	<0.0008
	X=	***0.016***	***<0.0008***
	s=	***0.001***	***-***
Euro 578-1	0.2500	0.017	0.0658
Ferro-Molybdenum	0.2488	0.018	0.0655
0.016% ±0.002% C	0.2493	0.018	0.0651
0.065% ±0.003% S	0.2536	0.019	0.0656
	0.2490	0.018	0.0656
	X=	***0.018***	***0.0655***
	s=	***0.001***	***0.0003***

**Results based upon linear, forced through origin calibrations utilizing EURO 577-1 Ferro-Vanadium @ 0.089% Carbon and EURO 578-1 Ferro-Molybdenum @ 0.065% Sulfur.*

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