Application Note

Instrument: TC600/TCH600

Oxygen and Nitrogen Determination in 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 most common ferroalloys consist of silicon, manganese, and chromium and are used as vehicles to get these alloying elements into the molten metal when making steel or cast iron. For example, silicon is used to deoxidize steel and is an alloying element in cast iron. Manganese is used as an alloying element and mitigates harmful effects of sulfur in cast iron and steel. Chromium increases corrosion resistance in stainless steels. Unfortunately, unwanted elements can also be part of these ferroalloys—such as oxygen, nitrogen, and hydrogen—and can impact the quality of the metal if not known and accounted for. Excess oxygen promotes unwanted oxide formation and can react with carbon to form carbon monoxide. This removes carbon that could be used to alloy, and the excess gas can cause porosity during solidification. Excess hydrogen can cause brittleness in solidified steel, and in extreme cases, can form blowholes where it escapes in larger amounts. Nitrogen will substantially decrease the ductility of steel, especially in high temperature zones, and is not easily removed. Subsequently, the determination of hydrogen, nitrogen, and oxygen levels in the ferroalloy feed stock is a critical quality control parameter.

Sample Preparation

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

Accessories

782-720 Graphite Crucibles, 780-721 Lower Electrode Tip for 782-720 Crucibles without automation, or 618-376 Lower Electrode Tip for 782-720 Crucibles with automation, 502-822 Nickel Capsules, 501-073 Graphite Powder, 766-053 Crucible Tweezers, 760-138 Sample Tweezers

Calibration Samples

LCRM®, LRM®, NIST, or other suitable reference materials. Ferroalloy, steel, or refractory metal reference materials can be used.*

**Multi-matrix calibrations are not recommended.*

Method Parameters

Analysis Parameters	
Outgas Cycles	3
Analysis Delay	20 s
Analysis Delay Comparator	1.000
Analysis Type	Semi-Auto Analysis
Auto Analyze on Mass Entry	Disabled
Pre-Analyze Crucible Outgas	Disabled

Element Parameters	*Oxygen*	*Nitrogen*
Minimum Analysis Time	45 s**	70 s**
Significant Digits	5	5
Conversion Factor	1.000000	1.000000
Integration Delay	5 s	15 s
Comparator Level	1.000000%	1.000000%
Stop if below (%)	0.000000	0.000000

***Instruments capable of using an argon carrier will require longer minimum analysis times to achieve full analyte recovery.*

Furnace Parameters

Furnace Control Mode	*Power*
Pre-Analyze Purge Time	—
Purge Time	10 s
Outgas Time	15 s
Outgas Cool Time	5 s
Outgas Low Power	5900 W†
Outgas High Power	5900 W†
Outgas Ramp Rate	—
Analyze Low Power	5000 W†
Analyze High Power	5000 W†
Analyze Ramp Rate	—
Sample Prep Time	—
Sample Prep Power	—
Temperature Sustain	None

†*May vary depending on line voltage. Level can be adjusted to facilitate recovery and/or reduce crucible burn-through. Instruments capable of using an argon carrier will require a reduced power setting. Typically, the furnace power setting is reduced 10-20% when performing analysis in argon.*

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Determine Blank.
   a. Enter a 1.0000 g mass into the Sample Login (F3) using Blank as the sample name.
   b. Press the loader switch on the front of the furnace. After a short delay, the loading head slide block will open.
   c. Place a 502-822 Nickel Capsule into the open port at the top of the loading head.

   Note: Use the same part number and lot number of capsules that will be used for the analysis of samples. During the weighing and introduction of the sample into the nickel capsule, the entire operation must be accomplished using clean tweezers only. Never touch the capsule with your fingers.

   d. Press the loader switch again. The loading head slide block will close and the furnace electrode will open.
   e. Clean the upper and lower electrode manually or with an equipped automatic cleaner.
   f. Add ~0.05 g of 501-073 Graphite Powder to a 782-720 Graphite Crucible.
   g. Place the crucible onto the electrode tip.

h. Press the loader switch, the electrode will close, and the analysis sequence will start and end automatically.
i. Repeat steps 2a through 2h a minimum of three times.
j. Set the blank following the procedure outlined in the operator's instruction manual.

3. Calibrate/Drift Correct.
   a. Weigh ~0.1 g of an appropriate refractory or ferroalloy reference material or ~0.5 g of a steel reference material into a 502-822 Nickel Capsule.
   b. Enter the mass and sample identification into the Sample Login (F3).
   c. Press the loader switch on the front of the furnace. After a short delay, the loading head slide block will open.
   d. Place the nickel capsule containing the reference material into the open port at the top of the loading head.
   e. Press the loader switch again, the loading head slide block will close and the furnace electrode will open.
   f. Clean the upper and lower electrode manually or with an equipped automatic cleaner.
   g. Add ~0.05 g of 501-073 Graphite Powder to a 782-720 Graphite Crucible.
   h. Place the crucible onto the electrode tip.
   i. Press the loader switch, the lower electrode will close and the analysis sequence will start and end automatically.
   j. Repeat steps 3a through 3i a minimum of three times for each calibration/drift reference material used.
   k. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.

4. Analyze Samples.
   a. Weigh ~0.10 g of a ferroalloy sample into a 502-822 Nickel Capsule.
   b. Enter mass and sample identification into Sample Login (F3).
   c. Press the loader switch on the front of the furnace. After a short delay, the loading head slide block will open.
   d. Place the nickel capsule containing the sample into the open port at top of the loading head.
   e. Press the loader switch again, the loading head slide block will close and the furnace electrode will open.
   f. Clean the upper and lower electrode manually or with an equipped automatic cleaner.
   g. Add ~0.05 g of 501-073 Graphite Powder to a 782-720 Graphite Crucible.
   h. Place the crucible onto the electrode tip.
   i. Press the loader switch, the lower electrode will close, and the analysis sequence will start and end automatically.
   j. Repeat steps 4a through 4i as necessary.

Typical Results†

Sample	*Mass (g)*	*% Oxygen*	*% Nitrogen*
BCS 204/4	0.1111	0.314	0.031
Ferro Chromium	0.0977	0.296	0.030
0.031% Nitrogen	0.1020	0.313	0.029
	0.0980	0.318	0.030
	0.0995	0.313	0.030
	Avg =	***0.311***	***0.030***
	s =	***0.008***	***0.001***

Sample	*Mass (g)*	*% Oxygen*	*% Nitrogen*
Ferro Manganese	0.1035	0.179	0.013
	0.0972	0.188	0.013
	0.0989	0.172	0.013
	0.0992	0.179	0.013
	0.1010	0.171	0.013
	Avg =	***0.178***	***0.013***
	s =	***0.007***	***0.0001***

Ferro Chromium	0.1026	0.124	0.068
Powder	0.1044	0.130	0.072
	0.1123	0.129	0.070
	0.1081	0.129	0.068
	0.0969	0.128	0.070
	Avg =	***0.128***	***0.070***
	s =	***0.002***	***0.002***

†*Results based upon linear, forced through origin calibrations utilizing LECO 502-876 Lot: 0316 Titanium LCRM (0.304% Oxygen) and LECO 502-904 Lot: 0599 Steel LCRM (0.0512% Nitrogen) using a Helium carrier.*

LECO Corporation | 3000 Lakeview Avenue | St. Joseph, MI 49085 | Phone: 800-292-6141 | 269-985-5496
info@leco.com • www.leco.com | ISO-9001:2015 Q-994 |

