Application Note

EMPOWERING RESULTS

Instrument: ON736

Determination of Oxygen and Nitrogen 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 or 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 and nitrogen - 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. Nitrogen will substantially decrease the ductility of steel, especially in high temperature zones, and is not easily removed. Subsequently, the determination of oxygen and nitrogen levels in the ferroalloy feed stock is a critical quality control procedure.

Instrument Model and Configuration

The ON736 is designed for simultaneous wide-range measurement of oxygen and nitrogen content. A pre-weighed sample is placed in a graphite crucible, which is heated in an impulse furnace to release analyte gases. Oxygen present in the sample reacts with the graphite crucible to form carbon monoxide (CO) and nitrogen is liberated as N_2. An inert carrier gas, helium or argon, sweeps the liberated analyte gases out of the furnace, through a mass flow controller and then through a heated reagent which converts CO to CO_2 and H_2 to H_2O. The gases then flow through a non-dispersive infrared (NDIR) cell to detect the oxygen as CO_2. The CO_2 and H_2O are then scrubbed out of the carrier gas stream leaving nitrogen as the only analyte gas remaining. A Thermal Conductivity (TC) detector is then used to detect nitrogen.

Sample Preparation

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

Note: LECO Reference Materials typically do not require preparation prior to analysis. Refer to preparation statement on the reference material certificate.

Accessories

782-720 Graphite Crucibles, 782-721 Lower Electrode Tip for 782-720 crucibles without automation, 618-376 Lower Electrode Tip for 782-720 crucibles with automation, 502-822 Nickel Capsules, 501-073 Graphite Powder, 501-598 Nibbled Nickel Flux, 766-053 Crucible Tweezers, and 760-138 Sample Tweezers.

Reference Materials

LCRM®, LRM®, NIST, or other suitable reference materials.

*Method Parameters**

General Parameters	
Carrier Gas Type	Helium or Argon
Sample Introduction	Automated Sample Drop
Analysis Delay	30 s
Wait for User to Load Sample	Yes
Vacuum On Time	18 s

Furnace Parameters	**Helium**	**Argon**
Furnace Control Mode	Power	Power

Outgas Parameter	**Helium**	**Argon**
Cycles	2	2
Power Mode	Constant	Constant
Power	5500** W	4500** W
Time	20 s	15 s
Cool Time	5 s	5 s

Analyze Furnace Settings, Step 1	**Helium**	**Argon**
Power Mode	Constant	Constant
Power	4800** W	3700** W

***May vary based on line voltage. Adjust to improve recovery or reduce crucible burn-through.*

	Helium		Argon	
Element Parameters	**Oxygen**	**Nitrogen**	**Oxygen**	**Nitrogen**
Integration Delay	5 s	15 s	5 s	15 s
Starting Baseline	2 s	2 s	2 s	2 s
Use Comparator	No	No	No	No
Integration Time	30 s	65 s	70 s	120 s
Use Endline	Yes	Yes	Yes	Yes
Ending Baseline	2 s	2 s	2 s	2 s

**Refer to 736 Series Operator's Instruction manual for parameter definitions.*

Procedure

1. Prepare the instrument as outlined in the operator's instruction manual.
2. Determine Blank.
   a. Log in a minimum of three Blank replicates in the Login screen.
   b. Weigh ~0.4 g of 501-598 Nibbled Nickel Flux into a 502-822 Nickel Capsule.
   c. Press the Analyze button on the instrument screen. After a short delay, the loading head slide-block will open.
   d. Place the nickel capsule containing the nibbled nickel flux into the open port at the top of the loading head.
   e. Press the Analyze button on the instrument screen again, and the loading head slide-block will close and the lower electrode will open.
   f. Clean the upper and lower electrodes either manually, or with an equipped automatic cleaner.
   g. Weigh ~0.05 g of 501-073 Graphite Powder into a 782-720 Graphite Crucible.
   h. Firmly place the graphite crucible containing the graphite powder on the lower electrode tip.
   i. Press the Analyze button on the instrument screen, and the lower electrode will close. The analysis sequence will start and end automatically.
   j. Perform steps 2b through 2i a minimum of three times.
   k. Set the blank following the procedure outlined in the operator's instruction manual.
3. Calibration/Drift Correction.
   a. Log in a minimum of three standard/drift replicates for each calibration/drift material being utilized in the Login screen.
   b. Weigh an appropriate mass of a calibration/drift material into a 502-822 Nickel Capsule and enter the mass and sample identification into the appropriate replicate fields in the Login screen.
   c. Add ~0.4 g of 501-598 Nibbled Nickel Flux to the nickel capsule, covering the calibration/drift material.
   d. Press the Analyze button on the instrument screen. After a short delay, the loading head slide-block will open.
   e. Place the nickel capsule containing the calibration/drift material and nibbled nickel flux into the open port at the top of the loading head.
   f. Press the Analyze button on the instrument screen again, and the loading head slide-block will close, and the lower electrode will open.
   g. Clean the upper and lower electrodes either manually, or with an equipped automatic cleaner.
   h. Weigh ~0.05 g of 501-073 Graphite Powder into a 782-720 Graphite Crucible.
   i. Firmly place the graphite crucible containing the graphite powder on the lower electrode tip.
   j. Press the Analyze button on the instrument screen, and the lower electrode will close. The analysis sequence will start and end automatically.
   k. Perform steps 3b through 3j a minimum of three times for each calibration/drift sample utilized.
   l. Calibrate/drift following the procedure outlined in the operator's instruction manual.
   m. Verify the calibration by analyzing several replicates of an appropriate mass of another/different calibration/drift material, following steps 3b through 3j, and verify that the results are within the tolerance range.
4. Analyze Samples.
   a. Log in the desired number of sample replicates in the Login screen.
   b. Weigh an appropriate mass (~0.10 – 0.14 g) of a ferroalloy sample into a 502-822 Nickel Capsule and enter the mass and sample identification into the appropriate replicate fields in the Login screen.
   c. Add ~0.4 g of 501-598 Nibbled Nickel Flux to the nickel capsule, covering the sample.
   d. Press the Analyze button on the instrument screen. After a short delay, the loading head slide-block will open.
   e. Place the nickel capsule containing the sample and nibbled nickel flux into the open port at the top of the loading head.
   f. Press the Analyze button on the instrument screen again, and the loading head slide-block will close, and the lower electrode will open.
   g. Clean the upper and lower electrodes either manually, or with an equipped automatic cleaner.
   h. Weigh ~0.05 g of 501-073 Graphite Powder into a 782-720 Graphite Crucible.
   i. Firmly place the graphite crucible containing the graphite powder on the lower electrode tip.
   j. Press the Analyze button on the instrument screen, and the lower electrode will close. The analysis sequence will start and end automatically.
   k. Perform steps 4b through 4j for each sample replicate being analyzed.

Typical Results†

Results are based on a linear, force through origin calibration utilizing 503-511 (Lot: 0800) LCRM Titanium Pins (0.365% Oxygen) for oxygen determination, and a linear, force through origin calibration utilizing 502-904 (Lot: 0599) LCRM Steel Pins (0.0738% Nitrogen) for nitrogen determination. The oxygen calibration was verified using 502-921 (Lot: 0596) LCRM Titanium Pins (0.066% Oxygen). The nitrogen calibration was verified using 502-921 (Lot: 0596) LCRM Titanium Pins (0.007% Nitrogen) and 502-999 (Lot: 1000) LCRM Steel Chip (0.0005% Nitrogen).

	Helium			Argon		
	Mass (g)	*% O*	*% N*	*Mass (g)*	*% O*	*% N*
BCS 203/5 Ferrochromium	0.1229	0.133	0.016	0.1204	0.159	0.015
0.017% N (Certified Value)	0.1225	0.118	0.016	0.1212	0.143	0.015
	0.1228	0.117	0.016	0.1205	0.138	0.017
	0.1222	0.135	0.016	0.1203	0.122	0.017
	0.1243	0.134	0.016	0.1204	0.123	0.018
	Avg =	***0.128***	***0.016***	***Avg =***	***0.137***	***0.016***
	s =	***0.009***	***<0.001***	***s =***	***0.015***	***0.001***
BCS 204/1 High Carbon Ferrochromium	0.1208	0.043	0.014	0.1187	0.045	0.015
	0.1205	0.049	0.015	0.1217	0.051	0.016
	0.1205	0.051	0.014	0.1196	0.059	0.015
	0.1197	0.050	0.014	0.1210	0.053	0.017
	0.1209	0.047	0.014	0.1200	0.053	0.016
	Avg =	***0.048***	***0.014***	***Avg =***	***0.052***	***0.016***
	s =	***0.003***	***<0.001***	***s =***	***0.005***	***0.001***
EURO 583-1 Ferromanganese	0.1195	0.128	0.036	0.1230	0.135	0.039
0.041% N (Informational Value)	0.1226	0.135	0.036	0.1236	0.134	0.039
	0.1230	0.124	0.034	0.1245	0.135	0.039
	0.1241	0.125	0.035	0.1234	0.140	0.037
	0.1238	0.124	0.033	0.1230	0.136	0.038
	Avg =	***0.127***	***0.035***	***Avg =***	***0.136***	***0.038***
	s =	***0.005***	***0.001***	***s =***	***0.002***	***0.001***
BCS 204/4 High Carbon Ferrochromium	0.1208	0.291	0.028	0.1201	0.332	0.029
0.031% N (Certified Value)	0.1190	0.302	0.027	0.1196	0.331	0.031
	0.1198	0.298	0.028	0.1189	0.310	0.031
	0.1226	0.295	0.029	0.1205	0.315	0.029
	0.1220	0.295	0.029	0.1212	0.310	0.030
	Avg =	***0.296***	***0.028***	***Avg =***	***0.320***	***0.030***
	s =	***0.004***	***0.001***	***s =***	***0.011***	***0.001***

†*Multi-matrix calibrations are not recommended for the determination of a single element.*

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