Application Note

Instrument: ONH836

Determination of Oxygen, Nitrogen, and Hydrogen in Iron, Steel, Nickel-base, and Cobalt-base Alloys: Comparison of Analytical Performance Between Argon and Helium Carrier

LECO Corporation; Saint Joseph, Michigan USA

Introduction

Oxygen, nitrogen, and hydrogen determination in iron, steel, nickel-base, and cobalt-base alloys are some of the most important quality metrics for these materials. Oxygen is used to create steel from pig iron by removing excess carbon. Oxygen content must be controlled to limit the amount of carbon monoxide that can be formed during solidification, which may cause excessive porosity. Nitrogen is considered both an impurity as well as an important alloying agent. It can be present as a nitride or interstitially in its gaseous form. Increased nitrogen content is known to increase yield and tensile strength, thus decreasing ductility and formability. Excessive levels may evolve during solidification, increasing porosity. High hydrogen content is the primary cause of embrittlement, blistering and flaking due to its high mobility through the lattice and provides no potential alloying benefits. The ONH836 utilizes a high-power electrode furnace to quickly and efficiently release the target gases from within the sample, which allows for a very rapid simultaneous determination of oxygen, nitrogen, and hydrogen.

Sample Preparation

Utilizing appropriate sampling and sample preparation techniques is important because traditional methods used to obtain samples for oxygen and nitrogen determination are different from those recommended for hydrogen determination, especially when sampling molten metal. The main difference between steel sampling procedures for oxygen/nitrogen and steel sampling procedures for hydrogen is due to the mobility of hydrogen. Special precautions must be taken when sampling for hydrogen. From molten steel and iron, a sample must be quickly quenched in cold water and chilled in a refrigerant such as liquefied nitrogen or a mixture of acetone and solid carbon dioxide in order to reduce loss of hydrogen by diffusion. Loss of oxygen and nitrogen by diffusion is not a problem. A sample that is taken for hydrogen determination and chilled in a refrigerant can also be used for oxygen and nitrogen determination. However, a sample that is taken for oxygen and/or nitrogen determination is typically not suitable for hydrogen determination due to hydrogen loss by diffusion. Surface contamination must be removed by filing or light grinding, using care not to overheat the sample. Subsequently, the prepared sample is washed in a suitable solvent such as acetone and dried with warm air. The prepared sample must be analyzed immediately after preparation. Chip and powder samples are typically analyzed on an as-received basis. ASTM E 1806 and ISO 14284 are sampling/sample preparation documents specific for steel and iron and are an excellent source of information.

Method Reference

ASTM E1019 - Standard Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Fusion Techniques.

Accessories

776-247 Graphite Crucibles, 611-351-182 Lower Electrode Tip for 776-247 Crucibles without automation; 611-351-181 Lower Electrode Tip for 776-247 Crucibles with automation, 502-822 Nickel Capsules, 766-053 Crucible Tweezers, 760-138 Sample Tweezers.

Reference Materials

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

Method Parameters

General Parameters	**Helium Carrier Gas**	**Argon Carrier Gas**
Sample Introduction	Automated Sample Drop	Automated Sample Drop
Analysis Delay	20 s	20 s
Wait for User to Load Sample	Yes	Yes
Vacuum On Time	10 s	10 s

Element Parameters	**Helium Carrier Gas**			**Argon Carrier Gas**		
Element	*Oxygen*	*Nitrogen*	*Hydrogen*	*Oxygen*	*Nitrogen*	*Hydrogen*
Integration Delay	0 s	10 s	10 s	0 s	15 s	10 s
Starting Baseline	2 s	2 s	2 s	2 s	2 s	2 s
Use Comparator	No	No	No	No	No	No
Integration Time	40 s	65 s	65 s	45 s	95 s	95 s
Use Endline	Yes	Yes	Yes	Yes	Yes	Yes
Ending Baseline	2 s	2 s	2 s	2 s	2 s	2 s
Range Select	Auto	-	-	Auto	-	-
Range Lower Limit	800	-	-	800	-	-
Range Upper Limit	950	-	-	950	-	-

Furnace Parameter	**Helium Carrier Gas**	**Argon Carrier Gas**
Furnace Control Mode	Power	Power

Outgas Parameter	**Helium Carrier Gas**	**Argon Carrier Gas**
Cycles	2	2
Power Mode	Constant	Constant
Power	5500* W	4600* W
Time	20 s	15 s
Cool Time	5 s	5 s

Analyze Furnace Settings, Step 1	**Helium Carrier Gas**	**Argon Carrier Gas**
Power Mode	Constant	Constant
Power	4800* W	4100* W
Approximate Cycle Time	3 min	3.75 min

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

Procedure – Solid Samples

1. Prepare the instrument as outlined in the operator's instruction manual.
2. Determine the instrument blank.
   a. Log in a minimum of three Blank replicates.
   b. Press the Analyze button on the instrument screen. After a short delay, the loading head slide-block will open.
   c. Press the Analyze button on the instrument screen again and the loading head slide-block will close and the lower electrode will open.
   d. Clean the upper and lower electrode either manually or with an equipped automatic cleaner.
   e. Firmly place a 776-247 Graphite Crucible on the lower electrode tip.
   f. Press the Analyze button on the instrument screen, and the lower electrode will close and the analysis sequence will start and end automatically.
   g. Repeat steps 2b through 2f a minimum of three times.
   h. Set the blank following the procedure outlined in the operator's instruction manual.
3. Instrument calibration/drift correction.
   a. Log in a minimum of three Standard replicates.
   b. Weigh ~1.0 g of a calibration/drift sample, enter the mass and sample identification into the appropriate replicate fields.

   Note: LECO Reference Materials do not require preparation. See preparation statement on the reference material certificate.

   c. Press the Analyze button on the instrument screen. After a short delay, the loading head slide-block will open.
   d. Place the calibration/drift sample 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 electrode either manually or with an equipped automatic cleaner.
   g. Firmly place a 776-247 Graphite Crucible on the lower electrode tip.
   h. Press the Analyze button on the instrument screen, and the lower electrode will close and the analysis sequence will start and end automatically.
   i. Repeat steps 3b through 3h a minimum of three times for each calibration/drift sample utilized.
   j. Calibrate/drift following the procedure outlined in the operator's instruction manual.
4. Analyze Samples.
   a. Log in a Sample with the appropriate number of replicates.
   b. Weigh ~1.0 g of a prepared sample and enter the mass and sample identification into the appropriate replicate fields.
   c. Press the Analyze button on the instrument screen. After a short delay, the loading head slide-block will open.
   d. Place the sample 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 electrode either manually or with an equipped automatic cleaner.
   g. Firmly place a 776-247 Graphite Crucible on the lower electrode tip.
   h. Press the Analyze button on the instrument screen, and the lower electrode will close and the analysis sequence will start and end automatically.
   i. Repeat steps 4b through 4h for each sample being analyzed.

Procedure – Chip/Powder Samples

Note: Chip and powder samples should be a uniform mesh size. Chip samples with particle sizes of +40 Mesh (420 microns) may be analyzed following the procedure outlined for analysis of solid samples. Chip samples with particles sizes of -40 Mesh must be analyzed following the procedure outlined below.

1. Prepare the instrument as outlined in the operator's instruction manual.
2. Determine the instrument blank.
   a. Log in a minimum of three blank replicates.
   b. Press the Analyze button on the instrument screen. After a short delay, the loading head slide-block will open.
   c. Place a 502-822 Nickel Capsule (leave capsule open) into the open port at the top of the loading head.
   d. Press the Analyze button on the instrument screen again and the loading head slide-block will close and the lower electrode will open.
   e. Clean the upper and lower electrode either manually or with an equipped automatic cleaner.
   f. Firmly place a 776-247 Graphite Crucible on the lower electrode tip.
   g. Press the Analyze button on the instrument screen, and the lower electrode will close and the analysis sequence will start and end automatically.
   h. Repeat steps 2b through 2g a minimum of three times.
   i. Set the blank following the procedure outlined in the operator's instruction manual.
3. Instrument calibration/drift correction.
   a. Log in a minimum of three Standard replicates.
   b. Weigh ~1.0 g of a calibration/drift sample into a 502-822 Nickel Capsule (leave capsule open), and enter the mass and sample identification into the appropriate replicate fields.

   Note: LECO Reference Materials do not require preparation. See preparation statement on the reference material certificate. Solid reference materials may be utilized to calibrate when chip or powder reference materials are not available.

   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 calibration/drift sample 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 electrode either manually or with an equipped automatic cleaner.
   g. Firmly place a 776-247 Graphite Crucible on the lower electrode tip.
   h. Press the Analyze button on the instrument screen and the lower electrode will close and the analysis sequence will start and end automatically.

i. Repeat steps 3b through 3h a minimum of three times for each calibration/drift sample utilized.
j. Calibrate/drift the following the procedure outlined in the operator's instruction manual.

4. Analyze Samples.
   a. Log in a Sample with the appropriate number of replicates.
   b. Weigh ~1.0 g of a sample into a 502-822 Nickel Capsule and enter the mass and sample identification into the appropriate replicate fields.
   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 sample 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 electrode either manually or with an equipped automatic cleaner.
   g. Firmly place a 776-247 Graphite Crucible on the lower electrode tip.
   h. Press the Analyze button on the instrument screen, and the lower electrode will close and the analysis sequence will start and end automatically.
   i. Repeat steps 4b through 4h for each sample being analyzed.

Typical Results

Results are based on a linear, forced through origin calibration utilizing 502-874 (Lot: 0676) Steel Pins @ 0.0366 ±0.0006% Oxygen, 502-928 (Lot: 0634) Steel Pins LCRM @ 0.0738 ±0.0018% Nitrogen, and 502-855 (Lot: 0718) Steel Pins @ 6.7 ppm Hydrogen.

	Helium				Argon			
	Mass (g)	*% O*	*% N*	*ppm H*	*Mass (g)*	*% O*	*% N*	*ppm H*
Steel LCRM	1.0036	0.0036	0.0509	4.5	1.0047	0.0040	0.0511	5.2
LECO 502-903	1.0027	0.0037	0.0518	4.6	1.0043	0.0037	0.0498	4.6
Lot: 0673	1.0028	0.0034	0.0508	4.8	1.0020	0.0040	0.0502	5.1
% O = 0.0040 ±0.0005	1.0014	0.0035	0.0509	4.0	1.0023	0.0036	0.0508	4.5
% N = 0.0509 ±0.0017	1.0046	0.0039	0.0508	4.8	1.0064	0.0038	0.0506	4.5
% H = 4.4 ±0.7 ppm	***Avg =***	***0.0036***	***0.0510***	***4.5***	***Avg =***	***0.0038***	***0.0505***	***4.8***
	s =	***0.0002***	***0.0004***	***0.4***	***s =***	***0.0002***	***0.0005***	***0.3***
Steel Chip LCRM	0.9967	0.0210	0.0002	0.3	1.0259	0.0213	0.0002	0.4
LECO 502-704	1.0258	0.0211	0.0002	0.5	1.0285	0.0219	0.0003	0.6
Lot: 1001	1.0007	0.0208	0.0003	0.7	1.0049	0.0213	0.0001	0.4
% O = 0.0216 ±0.0006	1.0171	0.0212	0.0002	0.6	1.0076	0.0211	0.0002	0.6
% N = 0.0002 ±0.0001	0.9973	0.0210	0.0002	0.4	1.0129	0.0213	0.0002	0.9
	Avg =	***0.0210***	***0.0002***	***0.5***	***Avg =***	***0.0214***	***0.0002***	***0.6***
	s =	***0.0001***	***< 0.0001***	***0.1***	***s =***	***0.0003***	***0.0001***	***0.2***
Steel Pin	1.0007	0.0062	0.0558	3.5	0.9947	0.0065	0.0555	4.0
	0.9955	0.0063	0.0561	3.6	1.0042	0.0063	0.0556	3.8
	0.9998	0.0061	0.0553	3.4	1.0001	0.0064	0.0553	3.7
	1.0012	0.0062	0.0559	3.5	0.9997	0.0062	0.0556	3.7
	1.0030	0.0062	0.0563	3.6	1.0078	0.0062	0.0558	3.5
	Avg =	***0.0062***	***0.0559***	***3.5***	***Avg =***	***0.0063***	***0.0556***	***3.7***
	s =	***< 0.0001***	***0.0004***	***0.1***	***s =***	***0.0001***	***0.0002***	***0.2***
Nickel/Iron/Niobium	0.9843	0.0179	0.0112	6.9	1.0176	0.0167	0.0114	6.9
Alloy Powder	1.0139	0.0172	0.0116	6.9	1.0118	0.0173	0.0122	7.3
	1.0227	0.0173	0.0114	7.0	0.9945	0.0171	0.0116	6.5
	1.0157	0.0170	0.0115	6.9	0.9921	0.0169	0.0113	6.9
	1.0088	0.0171	0.0113	7.1	1.0066	0.0171	0.0122	7.1
	Avg =	***0.0173***	***0.0114***	***6.9***	***Avg =***	***0.0170***	***0.0118***	***6.9***
	s =	***0.0004***	***0.0002***	***0.1***	***s =***	***0.0002***	***0.0004***	***0.3***

LECO Corporation | 3000 Lakeview Avenue | St. Joseph, MI 49085 | Phone: 800-292-6141 | 269-985-5496
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