Application Note

Instrument: ON736

Oxygen and Nitrogen in Solid Iron, Steel, Nickel-Base, and Cobalt-Base Alloys

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Introduction

Determining the amount of oxygen and nitrogen in steel is critical to process and quality control when making steel, nickel-base, and cobalt-base alloys. Oxygen is used to create steel from pig iron by removing excess carbon, and to remove impurities through oxidation. Oxygen content must be controlled to limit the amount of carbon monoxide that can form during solidification, which may cause excessive porosity. Oxygen content can also be correlated to the amount of residual oxides, or non-metallic inclusions present in the cast alloy. The amount of nitrogen in the alloy is indicative of the expected mechanical and corrosion properties of the alloy. As the nitrogen levels increase, so does the corrosion resistance, wear resistance, strength, and hardness. Too much nitrogen has been known to cause embrittlement, poor formability, and inconsistent mechanical properties. The ON736 provides a cost-effective and efficient means of simultaneously determining the amount of oxygen and nitrogen in steel, nickel-base, and cobalt-base alloys. The following application note outlines the hardware requirements, system settings, and expected performance of this test.

Sample Preparation

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. ASTM E 1806 and ISO 14284 are sampling/sample preparation documents specific for steel and iron, and are an excellent source of information.

Method ASTM E1019

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.

Reference Materials

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

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 using the +Blank icon.
   b. Press the Analyze icon on the instrument screen. After a short delay, the loading head slide-block will open.
   c. Press the Analyze icon on the instrument screen again, 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 graphite crucible on the lower electrode tip.
   f. Press the Analyze icon on the instrument screen, 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 using the +Standard icon.
   b. Weigh ~1.00 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 icon 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 icon on the instrument screen again, 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 graphite crucible on the lower electrode tip.
   h. Press the Analyze icon on the instrument screen, 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 Sample with the appropriate number of replicates using the +Sample icon.
   b. Weigh ~1.00 g of a prepared sample, enter mass and identification into the appropriate replicate fields.
   c. Repeat steps 3c through 3h for sample analysis.

Method Parameters

General Parameters	**Helium Method**		**Argon Method**	
Sample Introduction	Automated Sample Drop		Automated Sample Drop	
Analysis Delay	20 s		35 s	
Wait for User to Load Sample	Yes		Yes	
Vacuum On Time	7 s		7 s	
Element Parameters	*Oxygen*	*Nitrogen*	*Oxygen*	*Nitrogen*
Integration Delay	0 s	10 s	5 s	10 s
Starting Baseline	2 s	2 s	2 s	2 s
Use Comparator	No	No	No	No
Integration Time	30 s	55 s	35 s	85 s
Use Endline	Yes	Yes	Yes	Yes
Ending Baseline	2 s	2 s	2 s	2 s
Comparator Level	—	—	—	—
Minimum Integration Time	—	—	—	—
Maximum Integration Time	—	—	—	—
Furnace Parameters				
Furnace Control Mode	Power		Power	
Outgas Furnace Settings				
Cycles	2		2	
Power Mode	Constant		Constant	
Power	5500* W		4000* W	
Time	12 s		12 s	
Cool Time	5 s		5 s	
Analyze Furnace Settings				
Step 1 Power Mode	Constant		Constant	
Power	4800* W		3500* W	
Approximate Cycle Time	2.75 Minutes		3.75 Minutes	

**May vary, depending on line voltage. Level can be adjusted to facilitate recovery and/or reduce crucible burn-through.*

Procedure – Chip/Powder 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 using the +Blank icon.
   b. Press the Analyze icon 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 icon on the instrument screen again, 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 graphite crucible on the lower electrode tip.
   g. Press the Analyze icon on the instrument screen, 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 using the +Standard icon.
   b. Weigh ~1.00 g of a calibration/drift sample into a 502-822 Nickel Capsule, 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 icon 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 icon on the instrument screen again, 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 graphite crucible on the lower electrode tip.
   h. Press the Analyze icon on the instrument screen, 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 Sample with the appropriate number of replicates using the +Sample icon.
   b. Weigh ~1.00 g of a prepared sample into a 502-822 Nickel Capsule, enter mass and sample identification into the appropriate replicate fields.
   c. Repeat steps 3c through 3h for sample analysis.

*Typical Results**

	Helium Method			Argon Method		
Name	*Mass(g)*	*Oxygen %*	*Nitrogen %*	*Mass(g)*	*Oxygen %*	*Nitrogen %*
502-348	0.9941	0.0011	0.0005	0.9951	0.0011	0.0004
High Purity Steel	0.9924	0.0010	0.0005	0.9944	0.0011	0.0005
Oxygen: 0.0011 ± 0.0002 %	0.9928	0.0011	0.0004	0.9935	0.0012	0.0004
Nitrogen: 0.0004 ± 0.0001 %	0.9942	0.0010	0.0005	0.9948	0.0012	0.0004
	0.9929	0.0010	0.0005	0.9938	0.0012	0.0004
	Avg =	***0.0011***	***0.0005***	***Avg =***	***0.0012***	***0.0004***
	s =	***0.00005***	***0.00003***	***s =***	***0.00002***	***0.00005***
502-856	1.0041	0.0033	0.0665	1.0053	0.0033	0.0660
Steel Pin	1.0040	0.0032	0.0669	1.0044	0.0034	0.0665
Oxygen: 0.0032 ± 0.0006 %	1.0058	0.0033	0.0668	1.0029	0.0034	0.0664
Nitrogen: 0.0662 ± 0.0013 %	0.9961	0.0034	0.0675	1.0033	0.0034	0.0660
	1.0038	0.0032	0.0673	1.0029	0.0032	0.0677
	Avg =	***0.0033***	***0.0670***	***Avg =***	***0.0033***	***0.0665***
	s =	***0.0001***	***0.0004***	***s =***	***0.0001***	***0.0006***
502-855	1.0005	0.0124	0.0680	1.0068	0.0122	0.0683
Steel Pin	1.0019	0.0120	0.0681	1.0054	0.0120	0.0664
Oxygen: 0.0121 ± 0.0003 %	1.0020	0.0120	0.0680	1.0027	0.0120	0.0688
Nitrogen: 0.0673 ± 0.0012 %	1.0020	0.0120	0.0677	1.0041	0.0118	0.0677
	1.0016	0.0120	0.0685	1.0027	0.0118	0.0681
	Avg =	***0.0121***	***0.0681***	***Avg =***	***0.0120***	***0.0679***
	s =	***0.0002***	***0.0003***	***s =***	***0.0002***	***0.0009***
BCS 149/3 Iron Granules	1.0163	0.0626	0.0026	1.0003	0.0602	0.0027
	1.0095	0.0612	0.0026	1.0125	0.0601	0.0025
	0.9881	0.0616	0.0026	1.0008	0.0609	0.0025
	0.9988	0.0621	0.0024	1.0140	0.0614	0.0025
	0.9897	0.0628	0.0026	1.0042	0.0597	0.0026
	Avg =	***0.0620***	***0.0026***	***Avg =***	***0.0605***	***0.0026***
	s =	***0.0007***	***0.0001***	***s =***	***0.0007***	***0.0001***

**Note: Results based on a linear force through origin calibration utilizing LECO 502-874 (Lot #0676) Steel Pins @ 0.0366 ± 0.0006 % Oxygen and LECO 502-887 (Lot #0744) Steel Pins @ 0.229 ± 0.003 % Nitrogen.*

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