Determination of Oxygen and Nitrogen in Graphite and Calcined Coke

LECO Corporation; Saint Joseph, Michigan USA

Instrument: TC600 Series

Introduction

The determination of oxygen and nitrogen in graphite and calcined coke allows end users to confirm that the gas content of the graphite or calcined coke will not cause issues like the creation of unwanted pollutants or increased porosity in their final products. This is a key quality parameter in the steel industry where calcined coke is used as a recarburizer, and graphite is used as an electrode in electric arc furnaces. The LECO TC600 oxygen and nitrogen elemental analyzer can simultaneously determine the amount of oxygen and nitrogen in graphite and calcined coke with an emphasis on ease-of-use, accuracy, and precision.

This method is written for graphite and fully calcined coke samples which contain minimal volatile matter. ***Not applicable for coal or non-calcined green coke.***

Calibration Standard

LECO 502-868, 502-870, or other suitable calibration samples

Accessories

776-247 Graphite Crucible; 611-351-182 Electrode Tip (Used With the 776-247 Crucible); 782-720 Graphite Crucible; 782-721 Electrode Tip (Used With the 782-720 Crucible); 502-822 Nickel Capsule;
501-073 Graphite Powder

Sample Preparation

Solid samples should be crushed with a clean mortar pestle or other suitable device to uniform consistency. Sample should be dried at 125°C for one hour to remove residual moisture.

Method Parameters

Analysis Parameters	
Outgas Cycles	3
Analysis Delay	20 seconds
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	40 seconds	70 seconds
Significant Digits	5	5
Conversion Factor	1.000000	1.000000
Integration Delay	5 seconds	15 seconds
Comparator Level	1.000000%	1.00000%
Stop if below (%)	0.00000	0.00000

Gas Dose Parameters	*Oxygen*	*Nitrogen*
Minimum Gas Dose Time	N/A	N/A
Integration Delay	N/A	N/A
Comparator Level	N/A	N/A
Gas Dose Cycles	N/A	N/A

Furnace Parameters	
Furnace Control Mode	Power
Purge Time	15 seconds
Outgas Time	15 seconds
Outgas Cool Time	5 seconds
Outgas Low Current	6000 watts*
Outgas High Current	6000 watts*
Outgas Ramp Rate	—
Analyze Low Current	4800 watts*
Analyze High Current	4800 watts*
Analyze Ramp Rate	—
Sample Prep Time	—
Sample Prep Power	—
Temperature Sustain	None
Furnace on Time	30 seconds
Temperature Sustain	None

Peak Find Parameters	Oxygen	Hydrogen	Nitrogen
Look for Shoulders	No		
Peak Threshold	0.000000	0.000000	0.000000

Calibration Parameters	Oxygen	Hydrogen	Nitrogen
y = + 1.0x + 0 (default setting)			

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

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Determine Blank.
   a. Enter 1.0000 g mass into Sample Login (F3) using Blank as the sample name.
   b. Press Loader Switch on front of furnace; after a short delay the loading head slide block will open.
   c. Leaving the capsule open, place a clean 502-822 Nickel Capsule into an open port at top of loading head.
   d. Press Loader Switch again; the loading head slide block will close, and the lower electrode will open.
   e. Place a 782-720 Graphite Crucible containing ~0.05 g of 501-073 Graphite Powder or a 776-247Graphite Crucible without graphite addition on the lower electrode pedestal.
   f. Press Loader Switch; the lower electrode will close, and the analysis sequence will start and end automatically.

Inorganic Application Note

g. Repeat steps 2a through 2f a minimum of three times.
h. Set the blank following the procedure outlined in the operator's instruction manual.

Note: Use same part number and lot number of capsules that will be used for the analysis of samples, leave capsule open. 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.

3. Calibrate/Drift Correct.
   a. Weigh calibration sample into a 502-822 Nickel Capsule; enter mass and sample identification into Sample Login (F3) for calibration.
   b. Press Loader Switch on front of furnace; the loading head slide block will open.
   c. Leaving the capsule open, place a 502-822 Nickel Capsule with sample into an open port at top of loading head.
   d. Press Loader Switch again; the loading head slide block will close, and the lower electrode will open.
   e. Place a 782-720 Graphite Crucible containing ~0.05 g of 501-073 Graphite Powder or a 776-247 Graphite Crucible without graphite addition on the lower electrode pedestal.
   f. Press Loader Switch; the lower electrode will close, and the analysis sequence will start and end automatically.
   g. Repeat steps 3a through 3g a minimum of three times for each calibration/drift sample used.
   h. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.
4. Analyze Samples.
   a. Weigh ~0.05 g to 0.1 g of graphite or calcined coke sample into nickel capsule; enter mass and sample identification into Sample Login (F3).
   b. Press Loader Switch on front of furnace; the loading head slide block will open.
   c. Leaving capsule open, place a 502-822 Nickel Capsule with sample into an open port at top of loading head.
   d. Press Loader Switch again; the loading head slide block will close, and the lower electrode will open.
   e. Place a 782-720 Graphite Crucible containing ~0.05 g 501-073 Graphite Powder or a 776-247 Graphite Crucible without graphite addition on the lower electrode pedestal.
   f. Press Loader Switch; the lower electrode will close, and the analysis sequence will start and end automatically.

Typical Results

Sample	*Mass g*	*Oxygen %*	*Nitrogen %*
Graphite	0.9600	0.050	0.006
	0.1015	0.048	0.006
	0.1020	0.051	0.007
	0.1004	0.049	0.006
	0.0982	0.046	0.006
	Avg =	***0.049***	***0.006***
	s =	***0.0019***	***0.0002***
Calcined	0.1023	0.006	0.003
Petroleum	0.0984	0.004	0.003
Coke	0.0949	0.006	0.003
	0.0939	0.005	0.004
	0.0968	0.005	0.003
	Avg =	***0.005***	***0.003***
	s =	***0.0009***	***0.0003***

LECO Corporation | 3000 Lakeview Avenue | St. Joseph, MI 49085 | Phone: 800-292-6141 | Fax: 269-982-8977
info@leco.com • www.leco.com | ISO-9001:2008 HQ-Q-994 | *LECO is a registered trademark of LECO Corporation.*

