Application Note

Instrument: ON836* **Including: ONH836.*

EMPOWERING RESULTS

Determination of Oxygen and Nitrogen in Graphite and Carbon-Based Battery Materials

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Introduction

The determination of Oxygen and Nitrogen in Carbon-based battery materials is used in optimizing performance and ensuring reliability. Oxygen influences Lithium-ion intercalation and structural stability, affecting energy density and cycle life. Nitrogen, often introduced through doping, can enhance electronic conductivity, catalytic activity, and reaction kinetics. Accurate measurement of these elements is essential for quality control, process optimization, and advanced electrode material development in battery research and manufacturing. This is typically achieved using inert gas fusion combined with infrared and thermal conductivity detection, providing precise and efficient determination of these key elements.

Instrument Model and Configuration

The ON836 is designed for simultaneous dynamic-range measurement of Oxygen and Nitrogen content. A pre-weighed sample is placed in the instrument's automated sample drop mechanism and automatically transferred into a graphite crucible. The graphite crucible with sample is heated in an impulse furnace to release analyte gases. Oxygen present in the sample reacts with the graphite crucible to form CO and CO_2. An inert gas carrier (Helium or Argon) sweeps the liberated analyte gases out of the furnace, through a mass flow controller, then through a series of detectors. CO and CO_2 are detected using non-dispersive infrared (NDIR) cells. The gas then flows through a heated reagent where the CO is oxidized to form CO_2, and H_2 is oxidized to form H_2O. The gas continues through another NDIR cell where CO_2 is detected. 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. Reference materials should be prepared according to the preparation statement on the certificate.

Note: Please reference the appropriate Safety Data Sheets (SDS) for safe handling of all reference materials and samples.

Accessories

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

Note: A reduced sample mass (~0.05 g) may be required for samples with a high free-swelling index. When weighing sample masses ≤0.05 g, a five-place balance is required.

Reference Materials

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

Method Parameters**

General Parameters	
Carrier Gas Type	Helium
Sample Introduction	Automated Sample Drop
Analysis Delay	30 s
Auto Analyze on Mass Entry	No
Outgas Before Mass Entry	No
Outgas At End Of Analysis	No
Wait for User to Load Sample	Yes
Load Crucible After Analysis	No
Nominal Mass	1.0000 g
Vacuum On Time	5 s

Element Parameters	*Oxygen*	*Nitrogen*
Integration Delay	0 s	10 s
Starting Baseline	2 s	2 s
Use Comparator	No	No
Integration Time	40 s	90 s
Use Endline	Yes	Yes
Ending Baseline	2 s	2 s
Range Select	Auto	--
Range Lower Limit	1950	--
Range Upper Limit	2100	--

Furnace Parameters	
Furnace Control Mode	Power

Outgas Parameters	
Cycles	2
Power Mode	Constant
Power	5200[†] W
Time	20 s
Cool Time	5 s

Surface Oxide Removal Parameters	
Remove Surface Oxide	No

Temperature Sustain Settings	
Temperature Sustain Mode	None

Analyze Furnace Settings		
Step 1	Power Mode	Constant
	Power	4750[†] W

***Refer to 836 Series Operator's Instruction Manual for parameter definitions.*
[†]May vary based on the line voltage. Adjust to improve recovery or reduce crucible burn-through.

Procedure

1. Prepare the instrument as outlined in the operator's instruction manual.
2. Determine the blank.
   a. From the Analysis Screen, use the Login Bar to add three or more 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 into the open port at the top of the loading head.
   d. Press the Analyze button on the instrument screen again. The loading head slide-block will close, and the lower electrode will open.
   e. Clean the upper and lower electrodes 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. The lower electrode will close, and the analysis sequence will start and end automatically.
   h. Perform steps 2b through 2g a minimum of three times.
   i. Set the blank following the procedure outlined in the operator's instruction manual.
3. Calibrate or drift correct.
   a. From the Analysis Screen, use the Login Bar to add the desired number of calibration/drift replicates (minimum of three).
   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. 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 material into the open port at the top of the loading head.
   e. Press the Analyze button on the instrument screen again. 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. Firmly place a 776-247 Graphite Crucible on the lower electrode tip.
   h. Press the Analyze button on the instrument screen. The lower electrode will close, and the analysis sequence will start and end automatically.
   i. Perform 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.
   k. Verify the calibration by analyzing several replicates of an appropriate mass of another/different calibration/drift material, following steps 3b through 3h, and verify that the results are within the tolerance range.
4. Analyze the samples.
   a. From the Analysis Screen, use the Login Bar to add the desired number of sample replicates.
   b. Weigh an appropriate mass (~0.10 g) of a sample into a 502-822 Nickel Capsule and enter the mass and sample identification into the appropriate replicate fields in the Login screen.

   Note: A reduced sample mass (≤ 50 mg) may be required for samples with a high free-swelling index. When weighing samples at ≤ 50 mg, a five-place balance should be used.

   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. 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. Firmly place a 776-247 Graphite Crucible on the lower electrode tip.
   h. Press the Analyze button on the instrument screen. The lower electrode will close, and the analysis sequence will start and end automatically.
   i. Perform steps 4b through 4h for each sample replicate being analyzed.

Typical Results

Data was generated utilizing a linear, force through origin calibration for Oxygen using ~0.75 g (for high range calibration) and ~0.25 g (for low range calibration) of LECO 502-702 (Lot 1000) LCRM Iron Powder (1.10 % O) and a linear, force through origin calibration for Nitrogen using ~0.5 g of LECO 502-904 (Lot 0566) LCRM Steel Pin (0.0923 % N). The high range Oxygen calibration was verified using ~0.70 g of JK47A Iron Powder (0.69 % O). The low range Oxygen calibration was verified using ~1.0 g of LECO 503-514 (Lot 0742-1) LCRM Steel Pin (0.0023 % O). The Nitrogen calibration was verified using ~0.1 g of JK47A Iron Powder (0.0062 % N).

	Mass (g)	Oxygen (%)	Nitrogen (%)
Graphite Powder	0.1002	0.32	0.004
Thermo Fisher Scientific	0.0997	0.30	0.004
(385031000)	0.1000	0.30	0.005
	0.1005	0.30	0.005
	0.0997	0.30	0.004
	$\bar{x}$ =	***0.30***	***0.005***
	s **=**	***0.01***	***<0.001***
Conductive Expanded Graphite Powder[††]	0.05015[‡]	13.7	0.018
MSE Supplies (TIMCAL Timrex	0.04989[‡]	13.4	0.020
BNB90)(PO5036)	0.05001[‡]	15.0	0.020
	0.04976[‡]	14.0	0.019
	0.04987[‡]	15.2	0.016
	$\bar{x}$ =	***14.3***	***0.018***
	s **=**	***0.8***	***0.002***
Hard Carbon Powder	0.0995	6.18	0.227
MSE Supplies (PO0199)	0.1002	6.22	0.239
	0.1004	6.14	0.252
	0.0998	6.17	0.254
	0.1001	6.64	0.250
	$\bar{x}$ =	***6.27***	***0.244***
	s **=**	***0.21***	***0.011***
SiO_x/C Composite Silicon-Based	0.0999	0.58	0.058
Anode Powder	0.0999	0.56	0.061
MSE Supplies (PO0197)	0.1003	0.58	0.060
	0.1000	0.57	0.061
	0.1001	0.56	0.058
	$\bar{x}$ =	***0.57***	***0.060***
	s **=**	***0.01***	***0.001***
Mesocarbon Microbeads	0.1003	0.03	0.001
Synthetic Graphite Powder	0.1003	0.03	0.002
MSE Supplies (PO0120)	0.0998	0.03	0.001
	0.1001	0.02	0.002
	0.1003	0.03	0.002
	$\bar{x}$ =	***0.03***	***0.001***
	s **=**	***<0.01***	***<0.001***
Silicon Carbon Alloy (950)[††]	0.05003[‡]	10.6	0.194
MSE Supplies (PO8157)	0.04970[‡]	11.1	0.218
	0.05001[‡]	10.6	0.204
	0.04955[‡]	11.1	0.208
	0.04950[‡]	11.4	0.216
	$\bar{x}$ =	***11.0***	***0.208***
	s **=**	***0.4***	***0.010***

$\bar{x}$ = Sample Mean; s = Sample Standard Deviation
[††]Due to its high free-swelling index, this sample was analyzed using a reduced sample mass.
[‡]A five-place balance was used to weigh these sample replicates.

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