

Oxygen/Nitrogen Determination in Nitrides

LECO Corporation; Saint Joseph, Michigan USA

Instrument: TC600, TCH600

Introduction

The determination of oxygen and nitrogen in nitrides is a quick and direct measurement of nitride purity. Analysis results can be used for the purpose of quality control or material grading. For example, the three materials analyzed in this note—silicon nitride (Si_3N_4), titanium nitride (TiN), and boron nitride (BN)—have known ratios of nitrogen to Si/Ti/B based upon their composition. Differences from this accepted value may be classified as general contamination. The oxygen value provides more detailed information regarding possible oxide contamination.

Sampling and Sample Preparation

A representative and uniform sample is required for proper analysis. Powder or granular samples cannot be placed directly into the loading head. Therefore, samples are weighed into a tin capsule and inserted into a nickel basket which is subsequently transferred to the loading head for analysis using the Semi-Automatic mode.

Accessories

782-720 Graphite Crucibles, 501-059 Tin Capsules, 502-344 Nickel Baskets, and 501-073 Graphite Powder. *NOTE: LECO 502-344 Nickel Baskets and 501-059 Tin Capsules can be used directly from the bottle without additional cleaning.*

Calibration Samples

NIST SRM 8983 Silicon Nitride; or other suitable reference materials.

Method Parameters

Analysis Parameters

Outgas Cycles	3
Analysis Delay	20 seconds
Analysis Delay Comparator	1
Analysis Type	Semi-Auto Analysis
Auto Analyze on Mass Entry	Disabled
Preliminary Crucible Outgas	Disabled

Element Parameters	Oxygen	Nitrogen
Minimum Analysis Time	80 seconds	100 seconds
Significant Digits	5	5
Conversion Factor	1.00	1.00
Integration Delay	5 seconds	15 seconds
Comparator Level	1.00 %	1.00%

Furnace Parameters

Furnace Control Mode	Power
Purge Time	20 seconds
Outgas Time	15 seconds
Outgas Cool Time	5 seconds



Outgas Low Power	6000 Watts*
Outgas High Power	6000 Watts*
Outgas Ramp Rate	0 Watts/second
Analyze Low Power	1200 Watts*
Analyze High Power	5000 Watts*
Analyze Ramp Rate	72 Watts/second
Sample Prep Time	0 seconds
Sample Prep Power	0 Watts
Temperature Sustain	None

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

Procedure

- Determine Blank.
 - Enter 1.0000 g mass into Sample Login (F3) using Blank as sample name.
 - Press Loader Switch on front of furnace, after a short delay the loading head slide block will open.
 - Insert a 501-059 Tin Capsule (leave capsule open) into a 502-344 Nickel Basket and place into open port at top of loading head.
Note: Use same part number and lot number of capsules and baskets that will be used for the analysis of samples. During the weighing and introduction of the sample into the tin capsule the entire operation must be accomplished using clean tweezers only. Never touch the capsule or nickel basket with your fingers.
 - Press Loader Switch again. The loading head slide block will close and the furnace lower electrode will open.
 - Add ~0.05 g 501-073 Graphite Powder to a 782-720 Graphite Crucible.
 - Place crucible on lower electrode.
 - Press Loader Switch. The lower electrode will close and the analysis sequence will start and end automatically.
 - Repeat steps 1a through 1g a minimum of three times.
 - Set the blank following the procedure outlined in the operator's instruction manual.
- Calibrate/Drift Correct.
 - Weigh 0.02 to 0.06 g of calibration sample into a 501-059 Tin Capsule; enter mass and sample identification into Sample Login (F3). Insert the tin capsule (leave capsule open) into a 502-344 Nickel Basket.

- b. Press Loader Switch on front of furnace. After a short delay the loading head slide block will open.
 - c. Place capsule and basket into open port at top of loading head.
 - d. Press Loader Switch. The lower electrode will open.
 - e. Add ~0.05 g 501-073 Graphite Powder to a 782-720 Graphite Crucible.
 - f. Place crucible on lower electrode.
 - g. Press Loader Switch. The lower electrode will close and the analysis sequence will start and end automatically.
 - h. Repeat steps 2a through 2g a minimum of three times for each calibration/drift sample used.
 - i. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.
3. Analyze Samples.
- a. Weigh 0.02 to 0.06 g sample into a 501-059 Tin Capsule and enter mass and sample identification into Sample Login (F3). Insert the tin capsule (leave capsule open) into a 502-344 Nickel Basket.
 - b. Proceed as directed in steps 2b through 2g.

Typical Results*

Sample	Mass g	Oxygen %	Nitrogen %
SRM 8983	0.0416	1.20	39.2
Si ₃ N ₄	0.0410	1.19	39.4
@ 1.20% O [†]	0.0412	1.21	39.4
39.23% N [†]	0.0424	1.20	39.0
	0.0418	1.20	39.1
	X =	1.20	39.2
	s =	0.007	0.18

BN	0.0214	0.81	56.6
@ 56.43% N [†]	0.0258	0.79	56.3
	0.0221	0.78	55.6
	0.0226	0.78	56.1
	0.0198	0.78	56.2
	X =	0.79	56.2
	s =	0.013	0.36

TiN	0.0212	0.32	22.3
@ 22.6% N [†]	0.0201	0.34	22.6
	0.0414	0.36	22.5
	0.0415	0.35	22.2
	0.0674	0.35	22.3
	0.0698	0.35	21.9
	X =	0.35	22.5
	s =	0.014	0.48

*Based on single standard calibration with NIST SRM 8983 @ 1.20% O and 39.23% N

[†]Theoretical Value

LECO Corporation

3000 Lakeview Avenue • St. Joseph, MI 49085 • Phone: 800-292-6141 • Fax: 269-982-8977
 info@leco.com • www.leco.com • ISO-9001:2000 • No. FM 24045 • LECO is a registered trademark of LECO Corporation.