

Inorganic Application Note

Oxygen and Nitrogen in Nitride Material

Instrument

TC500, TC600

Calibration Standard

NIST 8983 (silicon nitride) or other suitable standards

Accessories

782-720 Graphite Crucible, 782-721 Electrode Tip
(used with the 782-720 Crucible),
501-059 Tin Capsule, 502-344 Nickel Basket

Sample Weight

~20 to 50 milligrams

Analysis Time

60 seconds (minimum)

Furnace Current

Outgas 6200 Watts, Analyze 5200 Watts

Analysis Mode

Automatic mode

Sample Preparation

Sample should be powders of uniform particle size

Analysis Parameters

Outgas Cycles:	3
Analysis Delay:	20 sec.
Analysis Delay Comparator:	1.0000
Analysis Type:	Auto
Minimum Analysis Time:	30 sec. O; 60 sec. N
Significant Digits:	5
Conversion Factor:	1.0000
Integration Delay:	5 sec. O; 15 sec. N
Comparator Level:	10.0
Furnace Control Mode:	Power
Purge Time:	15 sec.
Outgas Time:	15 sec.
Outgas Cool Time:	5 sec.
Outgas High/Low:	6200 Watts
Outgas Ramp Rate:	0 Watts
Analyze High/Low:	5200 Watts
Analyze Ramp Rate:	0 Watts
Sample Prep Time:	0 sec.
Sample Prep Power:	0 Watts
Temperature Sustain:	None



TC500/600

Method

1. Prepare instrument as outlined in the operator's instruction manual.
2. Determine the nickel basket and tin capsule blank.
 - a. Enter 1.000 gram weight into weight stack.
 - b. Press the LOADER CONTROL switch and place a clean folded 501-059 Tin Capsule (see note below) inside a 502-344 Nickel Basket and insert into sample load port.
 - c. Press the LOADER CONTROL switch and place a 782-720 Graphite Crucible on lower electrode. Press the LOADER CONTROL switch again. The furnace will close and the analysis will begin and end automatically.
 - d. Repeat steps 2a thru 2c a minimum of three times.
 - e. Enter blank using the Auto Blank procedure; refer to operator's instruction manual.

NOTE: *If the blanks on the capsules and baskets exceed 0.002% Oxygen, wash the capsules in acetone and dry with warm air and repeat steps 2a thru 2e. Handle capsules and baskets with clean tweezers only. Capsules are to be folded for all analysis, removing trapped air.*

3. Calibrate instrument as outlined in the operator's instruction manual.
4. To analyze samples.
 - a. Weigh sample (~20 to 50 milligrams) into a tared tin capsule. Carefully fold capsule, "squeezing" as much air from capsule as possible. Reweigh sample then enter weight into weight stack.
 - b. Place folded capsule into 502-344 Nickel Basket.
 - c. Press the LOADER CONTROL switch and place the capsule and basket containing sample into sample load port.
 - d. Press the LOADER CONTROL switch and place a 782-720 Graphite Crucible on lower electrode. Press the LOADER CONTROL switch again. The furnace will close and the analysis will begin and end automatically.

Typical Results

Name	Mass	% Oxygen	% Nitrogen
NIST 8983	0.0209	1.138	40.16
Si ₃ N ₄ @	0.0206	1.151	40.25
1.20% O ±0.14	0.0198	1.147	39.93
39.23% N ±1.06	0.0207	1.152	39.92
	0.0205	1.138	40.54
	Average	1.145	40.16
	Std. Dev.	0.007	0.26



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