Inorganic Application Note

Oxygen, Nitrogen, and Hydrogen Determination in Refractory Metals*

Instrument

TCH600

Sampling and Sample Preparation

Sampling and sample preparation of refractory metals such as titanium and zirconium is somewhat different from that of steel. Unlike steel samples, hydrogen is not as mobile in this group of materials; therefore, storage in liquid nitrogen or dry ice is not required. However, it is important to keep the sample cool when cutting or sectioning. Sample preparation for oxygen and nitrogen determination has been different from that for hydrogen determination. Typically, titanium and zirconium samples are chemically etched to remove surface contamination when oxygen and nitrogen are determined. However, etching can introduce hydrogen into the sample. ASTM method E 1409 "Determination of Oxygen and Nitrogen in Titanium and Titanium Alloys by the Inert Gas Fusion Technique", as updated in 1996, permits either etching or abrading (filing) of the test specimen. ASTM E 1937 "Determination of Nitrogen in Titanium and Titanium Alloys by the Inert Gas Fusion Technique" indicates that the test specimen be etched. ASTM E 1447 "Determination of Hydrogen in Titanium and Titanium Alloys by the Inert Gas Fusion Thermal Conductivity/Infrared Detection Method" permits surface preparation by abrading (if necessary to remove contamination). Differences in sample preparation present somewhat of a dilemma regarding simultaneous determination of O, N, and H in Titanium. However, abrading samples with a file to remove surface contamination will yield accurate O, N, and H results.

Accessories

776-247 Graphite Crucibles, 501-073 Graphite Powder, 502-344 Nickel Basket, 501-059 Tin Capsule (powder, chip, granular samples)

Note: LECO 502-344 Nickel Baskets are prepared using a proprietary procedure to ensure low and precise O, N and H content. These baskets can be used directly from the bottle without additional cleaning. To avoid contamination handle with clean forceps only.

Calibration Samples

Refractory metal reference materials (titanium, zirconium, etc.) from LECO, NIST or other suitable reference materials.

Sample Weight

0.1 to 0.15 g

**Refractory Metals include Ti, Zr, W, Mo, Ta, Nb, Hf, and their alloys.*

TCH600

Method Parameters

Analysis Parameters

Outgas Cycles	3
Analysis Delay	20 seconds
Analysis Delay Comparator	1.000
Analysis Type	Semi-Auto Analysis[1]

[1]In earlier software programs this is the same as Auto Analysis. Auto Analysis is now used for instruments equipped with auto-sample loading capability, refer to the latest version of the operator's instruction manual for additional details.

Element Parameters	**Oxygen**	**Nitrogen**	**Hydrogen**
Minimum Analysis Time	40 seconds	60 seconds	60 seconds
Significant Digits	5	5	6
Conversion Factor	1.00000	1.00000	1.00000
Integration Delay	5 seconds	15 seconds	10 seconds
Comparator Level	1.00000	1.00000	5.00000
Stop if below (%)	0.000000	0.000000	0.000000

Furnace Parameters

Furnace Control Mode	Power
Pre-Analyze Purge Time	—
Purge Time	10 seconds
Outgas Time	15 seconds
Outgas Cool Time	5 seconds
Outgas Low Power	6000 watts*
Outgas High Power	6000 watts*
Outgas Ramp Rate	—
Analyze Low Power	5200 watts*
Analyze High Power	5200 watts*
Analyze Ramp Rate	—
Sample Prep Time	—
Sample Prep Power	—
Temperature Sustain	None

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

Procedure—Solid Samples

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Determine Blank.
   a. Enter 1.0000 g weight into weight stack.
   b. Press Loader Switch on front of furnace, after a short delay the loading head slide block will open.
   c. Place a 502-344 Nickel Basket into 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. Add ~75 to 100 mg of 501-073 Graphite Powder to a 776-247 Graphite Crucible.
   f. Place crucible on electrode pedestal.
   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 five times.
   i. Set the blank following the procedure outlined in the operator's instruction manual.
3. Calibrate/Drift Correct.
   a. Weigh ~0.1 to 0.15 g of a calibration sample and enter weight into weight stack.
   b. Place sample into a 502-344 Nickel Basket.
   c. Press Loader Switch on front of furnace, the loading head slide block will open.
   d. Place nickel basket/sample into open port at top of loading head.
   e. Press Loader switch again, the loading head slide block will close and the lower electrode will open.

f. Add ~75 to 100 mg of 501-073 Graphite Powder to a 776-247 Graphite Crucible.
g. Place crucible on the electrode pedestal.
h. Press Loader Switch, the lower electrode will close and the analysis sequence will start and end automatically.
i. Repeat steps 3a through 3h a minimum of five times for each calibration sample used.
j. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.

4. Analyze Samples
   a. Weigh ~0.1 to 0.15 g sample and enter weight into weight stack.
   b. Proceed as directed in steps 3b through 3h.

Typical Results—Solid Samples

Sample	Weight g	O %	N %	H ppm
LECO	0.1143	0.269	0.0174	11
502-201	0.1138	0.267	0.0161	11
Titanium Pin	0.1139	0.266	0.0174	11
0.267% O	0.1142	0.266	0.0172	13
0.017%N	0.1146	0.269	0.0167	12
	0.1126	0.269	0.0173	12
	0.1149	0.265	0.0170	12
	0.1145	0.266	0.0172	12
	0.1140	0.267	0.0164	12
	0.1144	0.266	0.0171	12
	X =	***0.267***	***0.0170***	***12***
	s =	***0.0016***	***0.0004***	***0.5***
LECO	0.1086	0.131	0.0028	19
502-047	0.1111	0.131	0.0028	20
Zirconium Pin	0.1103	0.131	0.0029	20
0.13% O	0.1141	0.131	0.0028	20
	0.1110	0.130	0.0025	19
	0.1096	0.131	0.0029	20
	0.1125	0.131	0.0029	20
	0.1052	0.131	0.0027	19
	0.1003	0.131	0.0027	20
	0.1132	0.132	0.0029	20
	X =	***0.131***	***0.0028***	***20***
	s =	***0.0004***	***0.0001***	***0.5***

Procedure—Powder/Chip Samples

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Determine Blank.
   a. Enter 1.0000 g weight into weight stack.
   b. Press Loader Switch on front of furnace, after a short delay the loading head slide block will open.
   c. 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.
   d. Press Loader Switch again, the loading head slide block will close and the furnace lower electrode will open.
   e. Add ~75 to 100 mg of 501-073 Graphite Powder to a 776-247 Graphite Crucible.
   f. Place crucible on the furnace electrode pedestal.
   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 five times.
   i. Set blank following the procedure outlined in the operator's instruction manual.
3. Calibrate/Drift Correct.
   a. Weigh ~0.1 to 0.15 g refractory metal calibration sample into a 501-059 Tin Capsule and enter weight into weight stack. *Note: Calibration samples can be solid; they do not have to be powder or chip.*
   b. Press Loader Switch on front of furnace, after a short delay the loading head slide block will open.
   c. Insert capsule into 502-344 Nickel Basket and place into open port at top of loading head.
   d. Press Loader Switch again, the loading head slide block will close and the furnace lower electrode will open.
   e. Add ~75 to 100 mg of 501-073 Graphite Powder to a 776-247 Graphite Crucible.
   f. Place crucible on furnace electrode pedestal.
   g. Press Loader Switch, the lower electrode will close and the analysis sequence will start and end automatically.
   h. Repeat steps 3a through 3g a minimum of five times for each calibration sample used.
   i. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.
4. Analyze Samples.
   a. Weigh ~0.1 to 0.15 g sample into a 501-059 Tin Capsule and enter weight into weight stack.
   b. Proceed as directed in 3b through 3g.

Typical Results—Powder Samples

Sample	Weight g	O %	N %	H ppm
Tantalum	0.1055	0.206	0.0045	38
Powder	0.1076	0.207	0.0045	39
	0.1096	0.205	0.0040	37
	0.1020	0.205	0.0044	38
	0.1052	0.206	0.0048	39
	X =	***0.206***	***0.0045***	***38***
	s =	***0.001***	***0.0003***	***1***

Delivering the Right Results

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