Inorganic Application Note

Determination of Oxygen and Nitrogen in Reactive/Refractory Metals and Their Alloys*

Approval

ASTM E-1409 Oxygen Determination in Titanium and Titanium Alloys
ASTM E-1569 Oxygen Determination in Tantalum
ASTM E-1937 Nitrogen Determination in Titanium and Titanium Alloys

Sample Preparation

A clean representative sample is required in order to obtain optimum results. Solid samples can be leached in suitable acid or abraded with a clean file, rinsed in acetone, and dried with warm air. Refer to ASTM methods above for further details.

Accessories

782-720 Crucible; 782-721 Electrode Tip; 502-344 UHP Nickel Baskets; 501-073 Graphite; 503-032 Glass Accelerator Scoop; 501-059 Tin Capsules—for powdered samples. (Additional steps required for powdered samples are noted in parenthesis.)

Calibration Standard

LECO 501-320 Titanium Pin, 502-047 Zirconium Pin, NIST, or other suitable reactive/refractory metal standard.

Method Parameters

Analysis Parameters

Outgas Cycles	2
Analysis Delay (seconds)	20
Analysis Comparator	1
Analysis Type	Auto

Element Parameters	**Oxygen**	**Nitrogen**
Minimum Time (seconds)	35	55
Conversion Factor	1.0	1.0
Integration Delay (seconds)	5	15
Comparator Level (%)	1	1

Furnace Parameters

Furnace Control Mode	Power
Purge Time (seconds)	15
Outgas Time (seconds)	20
Cool Time (seconds)	5
Outgas Power (Watts)	6300
Analyze Power (Watts)	5300

**This includes Ti, Zr, W, Mo, Ta, Nb, Hf, and their alloys.*

TC600

Typical Results

Titanium Pin	*Mass (g)*	*Oxygen (%)*	*Nitrogen (%)*
	0.1136	0.1826	0.0191
	0.1137	0.1826	0.0190
	0.1135	0.1828	0.0191
	0.1136	0.1841	0.0192
	0.1140	0.1829	0.0186
	0.1132	0.1861	0.0185
	0.1138	0.1846	0.0184
	0.1116	0.1836	0.0189
	0.1132	0.1836	0.0187
	0.1131	0.1836	0.0188
	Average	**0.1836**	**0.0188**
	Std. Dev.	**0.0011**	**0.00029**

Zirconium Wire	*Mass (g)*	*Oxygen (%)*	*Nitrogen (%)*
	0.0936	0.1238	0.0021
	0.1034	0.1235	0.0021
	0.0964	0.1225	0.0020
	0.1081	0.1236	0.0020
	0.1115	0.1245	0.0020
	0.1076	0.1263	0.0019
	0.1011	0.1244	0.0021
	0.1026	0.1239	0.0019
	0.1034	0.1237	0.0019
	0.0951	0.1236	0.0020
	Average	**0.1240**	**0.0020**
	Std. Dev.	**0.0010**	**0.00008**

Procedure

1. Determine the blank as follows:
   a. Enter the "blank" ID code with a 1.0000 g weight in the weight stack.
   b. Press the loader control switch, the sample loader will open.
   c. Place one 502-344 UHP Nickel basket into the loading head using clean tweezers. (Place a 501-059 Tin capsule into the Nickel basket before placing it in the loading head.)
   d. Press the loader control switch, the sample loader will close and seal and the furnace electrode will open.
   e. Remove crucible from electrode tip and discard. Clean furnace area using the appropriate brushes. Vacuum away loose dust.
   f. Place ~0.05 g 501-073 into the bottom of a 782-720 crucible. ~0.05 g is approximately a ¼ full 503-032 glass accelerator scoop.
   g. Place the crucible on the lower electrode.
   h. Press the loader control switch, the furnace electrode will close and the analysis sequence will start automatically.
   i. Repeat steps 1a through 1h at least four more times.
   j. Enter blank following routine outlined in operator's instruction manual.
2. Calibrate the instrument as follows:
   a. Weigh the calibration sample. (Weigh the calibration sample into the tin capsule.)
   b. Enter the calibration sample ID code and sample weight in the weight stack.
   c. Place the calibration sample (capsule) into a nickel basket.
   d. Press the loader control switch, the sample loader will open.
   e. Carefully place the calibration sample/nickel basket (capsule) into the loading head using clean tweezers. Make sure that the calibration sample (capsule) stays in the basket and the basket stays upright.
   f. Press the loader control switch, the sample loader will close and seal and the furnace electrode will open.

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Procedure (continued from page 2)

g. Remove crucible from electrode tip and discard. Clean furnace area using the appropriate brushes. Vacuum away loose dust.
h. Place ~0.05 g graphite into the bottom of a crucible.
i. Place the crucible on the lower electrode.
j. Press the loader control switch, the furnace electrode will close and the analysis sequence will start automatically.
k. Repeat steps 2a through 2j a minimum of three times.
l. Complete a calibration by following the auto calibration procedure as outlined in the operator's instruction manual.
m. Verify the calibration by analyzing the calibration sample again. It should fall within the expected tolerances. If not, repeat steps 2a through 2l again.

3. Analyze the samples as follows:
   a. Weigh ~0.1 g sample. (Weigh the sample into the tin capsule.)
   b. Enter the sample ID code and sample weight in the weight stack.
   c. Place the sample (capsule) into a nickel basket.
   d. Press the loader control switch, the sample loader will open.
   e. Carefully place the sample/nickel basket (capsule) into the loading head using clean tweezers. Make sure that the sample (capsule) stays in the basket and the basket stays upright.
   f. Press the loader control switch, the sample loader will close and seal and the furnace electrode will open.
   g. Remove crucible from electrode tip and discard. Clean furnace area using the appropriate brushes. Vacuum away loose dust.
   h. Place ~0.05 g graphite into the bottom of a crucible.
   i. Place the crucible on the lower electrode.
   j. Press the loader control switch, the furnace electrode will close and the analysis sequence will start automatically.

Theory of Operation

The TC600 is a microprocessor-based, software-controlled instrument that measures both nitrogen and oxygen in a wide variety of metals, refractories, and other inorganic materials. The inert gas fusion principle is employed. A weighed sample, placed in a high purity graphite crucible, is fused under a flowing helium gas stream at temperatures sufficient to release oxygen, nitrogen, and hydrogen. The oxygen in the sample combines with the carbon from the crucible forming primarily carbon monoxide (CO). In some instances, depending upon sample type and furnace temperature, some oxygen can be released directly as carbon dioxide (CO_2). The nitrogen present in the sample releases as molecular nitrogen, and any hydrogen present is released as hydrogen gas.

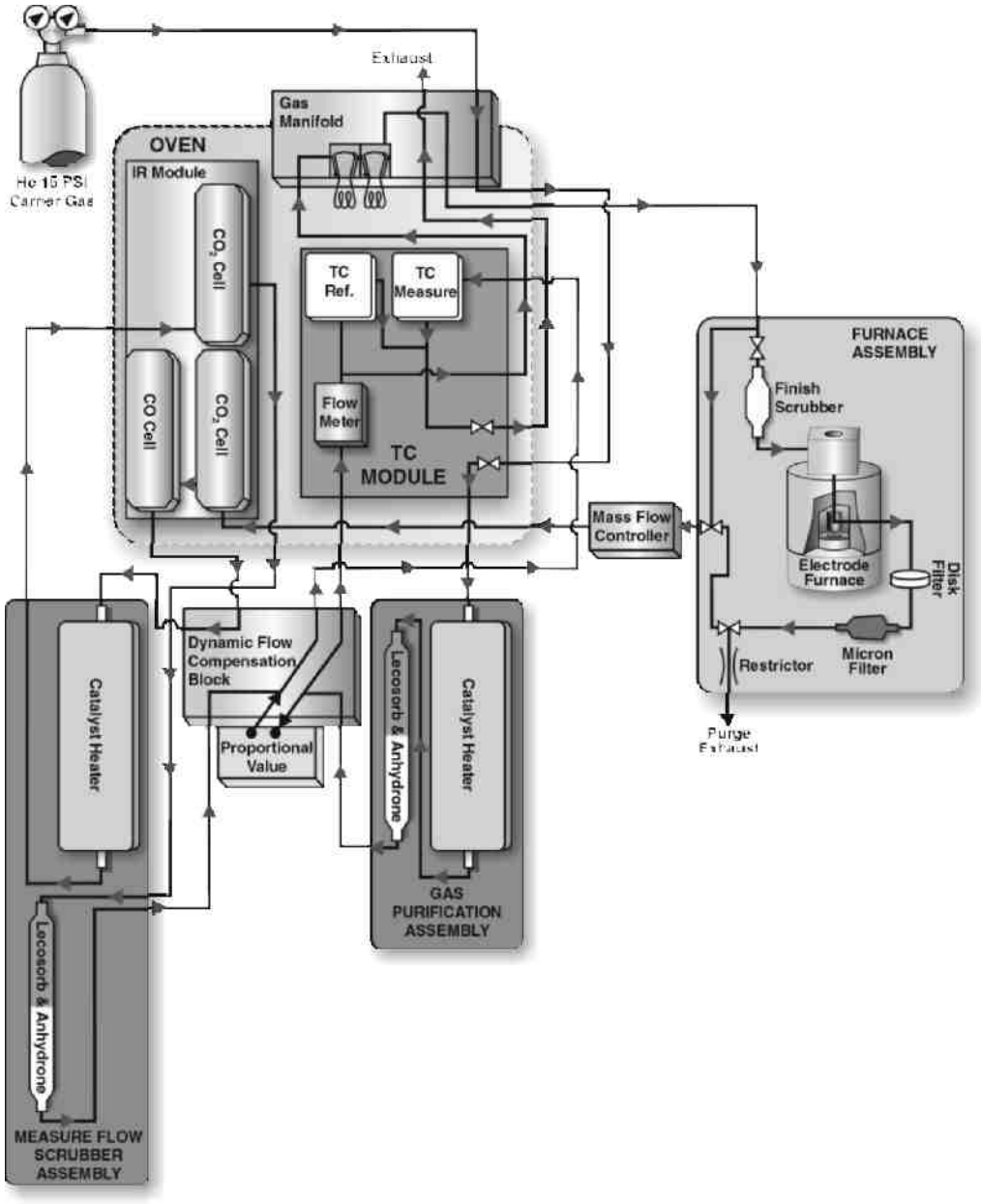
Oxygen Measurement

Oxygen is measured by infrared absorption. Sample gases first enter the IR module and pass through CO and CO_2 detectors. Oxygen present as either CO or CO_2 is detected. Following this, sample gas is passed through heated rare earth copper oxide to convert CO to CO_2 and any hydrogen to water. Gases then re-enter the IR module and pass through a separate CO_2 detector for total oxygen measurement. This configuration maximizes performance and accuracy for both low and high range. The instrument automatically chooses the optimum detection range.

Nitrogen Measurement

Nitrogen is measured by thermal conductivity (TC). Sample gases pass through heated rare earth copper oxide which converts CO to CO_2 and hydrogen to water. CO_2 and water are then removed with a Lecosorb/Anhydrone trap to prevent detection by the TC cell. Gas flow then passes through the TC cell for nitrogen detection.

TC600 Flow Diagram



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