Inorganic Application Note

Ultra Low Nitrogen and Oxygen in Iron, Steel, Nickel-Base, and Cobalt-Base Alloys

Approval ASTM E-1019

Sample Preparation

Surface contamination on the sample can cause significant errors in the analytical data; therefore, care must be taken to ensure a clean, representative sample is analyzed. See ASTM reference E-1806. Solid samples should be abraded with a clean file, rinsed in acetone, and dried with warm air prior to analysis. Samples that cannot be abraded due to irregular shapes (i.e. ball bearings), should be cleaned by alternate methods (i.e. solvent cleaning and/or etching). Care must be taken to remove all traces of solvent.

Accessories

782-720 Graphite Crucibles, 782-721 Electrode Tip

Calibration Standard

NIST, JSS, LECO, or other suitable calibrant

Method Parameters

Analysis Parameters

Outgas Cycles	2
Analysis Delay	25 seconds
Analysis Delay Comparator	1.0
Analysis Type	Auto Analysis

Element Parameters	**Oxygen**	**Nitrogen**
Minimum Analysis Time	35 seconds	55 seconds
Significant Digits	6	6
Conversion Factor	1.0	1.0
Integration Delay	0 seconds	15 seconds
Comparator Level	0.2	1.0

Furnace Parameters

Furnace Control Mode	Power
Purge Time	15 seconds
Outgas Time	20 seconds
Outgas Cool Time	8 seconds
Outgas Low Power	6000 watts
Outgas High Power	6000 watts
Analyze Low Power	5000 watts
Analyze High Power	5000 watts

TC600

Procedure

1. Prepare the instrument as outlined in the operator's instruction manual.
2. Determine the blank.
3. Calibrate as outlined in the operator's instruction manual.
4. To analyze samples:
   a. Weigh ~1.0 g of a prepared sample and enter the weight.
   b. Continue with analysis as outlined in the operator's instruction manual.

Typical Results

UHP Nickel Pin	Mass (g)	Oxygen (ppm)	Nitrogen (ppm)
	1.0001	4.04	0.81
	1.0004	3.84	0.69
	0.9999	4.25	0.85
	1.0002	4.22	0.78
	1.0008	3.93	0.94
	Average	**4.06**	**0.81**
	Std. Dev.	**0.18**	**0.09**

JSS GS-6a Ball Bearing Steel; 3.4 ppm O	Mass (g)	Oxygen (ppm)
	1.0440	3.57
	1.0439	3.50
	1.0438	3.52
	1.0439	3.68
	1.0438	3.60
	Average	**3.57**
	Std. Dev.	**0.07**

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, in all forms present, combines with the carbon from the crucible to form carbon monoxide. 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.

Delivering the Right Results

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