Inorganic Application Note

Oxygen and Nitrogen Analysis in Calcium Fluoride

Sample Preparation

Contamination of the sample can cause significant errors in the analytical data, therefore care must be taken to ensure a clean representative sample is analyzed.

Accessories

782-720 Crucible, 782-721 Electrode Tip, 502-344 UHP Nickel Baskets, 501-073 Graphite, 503-032 Glass Accelerator Scoop, 617-716 Quick Disconnect Tube, 775-306 Screen Filter, 501-081 Glass Wool, 502-351 Halogen Scrubber/Trap, (501-059 Tin Capsules for powdered samples); (Steps required for powdered and chip samples are contained within parenthesis).
NOTE: Tin capsules can also be used when analyzing solid samples.

Calibration Standard

LECO 501-644, LECO 501-550, NIST steel, or other suitable standard

Sample Weight

~0.1 g CaF_2; 0.5 to 1.0 g for steel calibration standards

Analysis Parameters

Outgas Cycles	2
Analysis Delay	30
Analysis Comparator	1
Analysis Type	Auto

Element Parameters

	Oxygen	*Nitrogen*
Minimum Time (sec.)	35	55
Integration Delay (sec.)	5	15
Comparator Level (%)	1	1

Furnace Parameters

Furnace Control Mode	Current
Purge Time (sec.)	15
Outgas Time (sec.)	20
Cool Time (sec.)	5
Outgas Low Power (Amps)	1050
Outgas High Power (Amps)	1050
Analyze Low Power (Amps)	975
Analyze High Power (Amps)	975

TC600

Halogen Trap Packing

1. Pack 1/2 inch of 501-081 Glass Wool into the flared bottom of the 617-716 Quick Disconnect Tube. Do not twist the glass wool and do not leave strands of glass wool in the neck of the tube.
2. Insert a 775-306 Screen Filter into the bottom of the quick disconnect tube. Position the o-ring towards the bottom and push the screen filter up into the quick disconnect tube 1/4 inch.
3. Fill the tube with 502-351 Halogen Scrubber and gently tap it down, leaving 1/2 inch free at the top of the tube.
4. Pack 1/2 inch of glass wool into the flared top of the tube.
5. Remove the 502-374 Particle Filter and install the quick disconnect tube in its place—note that the screen filter should be at the top of the tube.

   NOTE: Conditioning the trap. It is necessary to analyze 2 to 5 CaF_2 samples, in order to condition the trap before setting the blank and calibrating. Refer to step 3 in the method for instructions on analyzing samples.

Method

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 Graphite into the bottom of a 782-720 Crucible. ~0.5 g is approximately a 1/4 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 two 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 (capsule)/nickel basket 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.
   g. Remove the crucible from the 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 at least two more 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 (capsule)/nickel basket 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 the crucible from the 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.

Typical Results

Sample	**Weight**	**ppm O**	**ppm N**
CaF_2	0.1129	13.9	1.2
Crystal	0.1009	10.2	1.4
	0.1116	9.6	0.9
	0.1143	5.7	1.0
	Average	***9.9***	***1.1***
	Std. Dev.	***3.4***	***0.2***
CaF_2	0.1037	39.8	27.1
Pressed Powder	0.1043	32.2	26.3
(Broken into	0.1007	26.5	27.0
small chunks	0.1149	29.2	27.2
for analysis)	0.1014	20.6	25.6
	0.1123	24.1	27.7
	0.1093	29.4	25.1
	0.1131	25.3	28.3
	Average	***28.4***	***26.8***
	Std. Dev.	***5.8***	***1.1***

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.

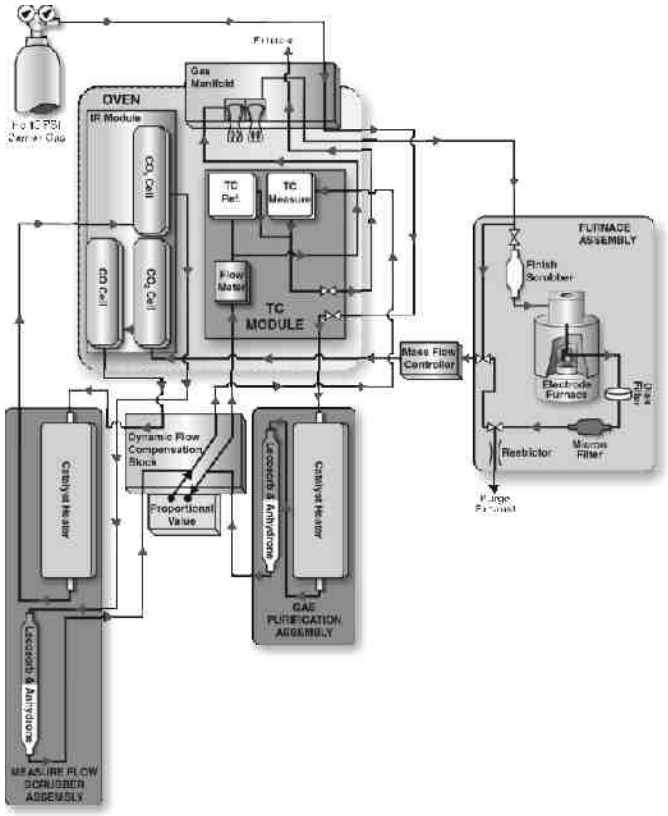
Oxygen Measurement

Oxygen is measured by infrared absorption (IR). 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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