Inorganic Application Note

Oxygen, Nitrogen and Hydrogen Determination in Steel and Iron

Instrument

TCH600

Sampling and Sample Preparation

Sampling and sample preparation is an important issue because traditional methods used to obtain samples for oxygen and nitrogen determination are different from those recommended for hydrogen, especially when sampling molten metal. The main difference in steel sampling procedures for oxygen/nitrogen and hydrogen is due to the mobility of hydrogen. Special precautions must be used when sampling for hydrogen. From molten steel and iron, a sample must be quickly quenched in cold water and chilled in a refrigerant such as liquefied nitrogen or a mixture of acetone and solid carbon dioxide in order to reduce losses of hydrogen from diffusion. Losses of oxygen and nitrogen from diffusion are not a problem. A sample that is taken for hydrogen and chilled in a refrigerant can also be used for oxygen and nitrogen determination. However, a sample that is typically taken for oxygen and/or nitrogen determination is not suitable for hydrogen determination due to hydrogen loss [diffusion]. Surface contamination must be removed by filing or light grinding using care not to overheat the sample. Subsequently the prepared sample is washed in a suitable solvent such as acetone and dried with warm air. The prepared sample must be analyzed immediately after preparation. ASTM E 1806 and ISO 14284 are sampling/sample preparation documents specific for steel and iron and are an excellent source of information.

Accessories

776-247 Graphite Crucibles; 782-720S Crucibles (can be used for high-oxygen iron/steel powder samples using Manual Top Load Procedure); 617-997 Funnel (for Manual Top Load Procedure)

Calibration Samples

LECO 502-416 or 502-457 One-Gram Steel Pins with O, N, and H contents determined; NIST or other suitable reference materials

Method Parameters

Analysis Parameters

Outgas Cycles	3
Analysis Delay	20 seconds
Analysis Delay Comparator	1.000
Analysis Type	Semi-Auto Analysis[1]
Auto Analyze on Mass Entry	Disabled
Pre-Analyze Crucible Outgas	Disabled

[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.*

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Element Parameters	Oxygen	Nitrogen	Hydrogen
Minimum Analysis Time	40 seconds	60 seconds	60 seconds
Significant Digits	5	5	6
Conversion Factor	1.000000	1.000000	1.000000
Integration Delay	5 seconds	15 seconds	10 seconds
Comparator Level	1.000000 %	1.000000 %	5.000000 %
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	5000 watts*
Analyze High Power	5000 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 gram weight into weight stack.
   b. Press Loader Switch on front of furnace, after a short delay the loading head slide block will open.
   c. Press Loader Switch again, the loading head slide block will close and the lower electrode will open.
   d. Place crucible on electrode pedestal.
   e. Press Loader Switch, the lower electrode will close and the analysis sequence will start and end automatically.
   f. Repeat steps 2a through 2e a minimum of five times.
   g. Set the blank following the procedure outlined in the operator's instruction manual.
3. Calibrate/Drift Correct.
   a. Weigh ~1.0 gram of a calibration sample and enter weight into weight stack.
   b. Press Loader Switch on front of furnace, the loading head slide block will open.
   c. Place sample 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. Place crucible on the electrode pedestal.
   f. Press Loader Switch, the lower electrode will close and the analysis sequence will start and end automatically.
   g. Repeat steps 3a through 3f a minimum of five times for each calibration sample used.
   h. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.
4. Analyze Samples.
   a. Weigh ~1.0 gram sample and enter weight into weight stack.
   b. Proceed as directed in steps 3b through 3f.

Typical Results—Solid Samples

Sample	Weight g	O ppm	N ppm	H ppm
LECO	1.0026	30	447	3.0
502-416	1.0006	31	454	3.2
Steel Pin	1.0024	28	448	3.3
29 ppm O	1.0013	30	453	3.3
450 ppm N	1.0005	29	452	3.2
3.2 ppm H	1.0004	30	454	3.1
	1.0007	29	453	3.1
	1.0034	30	456	3.2
	1.0008	29	451	3.1
	0.9989	30	451	3.3
	***X* =**	***30***	***451***	***3.2***
	***s* =**	***1***	***3***	***0.1***
LECO	1.0131	216	32	1.0
502-457	1.0116	215	33	1.1
Steel Pin	1.0127	217	33	1.1
213 ppm O	1.0132	217	33	0.9
33 ppm N	1.0115	217	33	1.2
0.9 ppm H	1.0117	212	31	1.0
	1.0120	216	32	1.2
	1.0129	217	32	0.8
	1.0117	217	32	1.3
	1.0125	209	31	0.7
	***X* =**	***215***	***32***	***1.0***
	***s* =**	***3***	***1***	***0.2***

Alternate Procedure—Powders/Chip Samples

If powder or chip samples are to be analyzed, they cannot be placed directly in the loading head. Options include weighing samples into capsules. Both tin and nickel capsules are available from LECO. There are issues related to blank, limited volume/sample weight and the increased time and manipulation to weigh samples in a capsule. In addition, using tin capsules can result in nitrogen recovery problems. There are two methods of manually loading a sample available to the TCH600 user.

Manual Analysis—The electrodes are opened after outgas and the sample is inserted into the crucible.

Manual Top Load—After outgas, loading head is opened and the sample is dropped into the crucible. For powder samples, a LECO 617-997 funnel can be inserted through the loading head into the crucible and the sample is transferred to the crucible via the funnel.

The preferred alternative is to use the Manual Top Load function of the TCH600. This option limits the outgassed crucible exposure to the atmosphere, reducing blank variability, subsequently improving precision.

Manual Top Load Procedure

1. Set Method Parameters as noted above with the following exceptions.
   a. Under *Analysis Parameters* set Analysis Type to Manual Top Load.
   b. Under *Furnace Parameters* set Pre-Analysis Purge Time to 50 seconds.
   c. Under *Element Parameters* set Minimum Analysis Time for oxygen to 45 seconds.
   d. Under *Element Parameters* set Comparator Level for Oxygen to 100%.
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 lower electrode will open.
   c. Place crucible on electrode pedestal.
   d. Press Loader Switch, the lower electrode will close and the outgas sequence will start automatically.
   e. When the outgas sequence is complete, an Add Sample message will appear in the lower left-hand corner of the instrument display. Press the Loader Switch and the loading head slide block will open.
   f. Place the 617-997 funnel into the open loading head.
   g. Remove the funnel, press the Loader Switch, the loading head slide block 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 ~1.0 gram of a calibration sample and enter weight into weight stack.
   b. Press Loader Switch on front of furnace, after a short delay the lower electrode will open.
   c. Place crucible on electrode pedestal.
   d. Press Loader Switch, the lower electrode will close and the outgas sequence will start automatically.
   e. When the outgas sequence is complete, an Add Sample message will appear in the lower left-hand corner of the instrument display. Press the Loader Switch and the loading head slide block will open.
   f. Place the 617-997 funnel into the open loading head and add sample taking care to make sure that all of the sample material is transferred into crucible.
   g. Remove funnel, press Loader Switch, the loading head slide block 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/drift sample used.
   i. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.
4. Analyze Samples.
   a. Weigh ~1.0 gram of sample and enter weight into weight stack.
   b. Proceed as directed in steps 3b through 3g.

Typical Results Manual Top Load Procedure—Powder Sample

Sample	Weight	O %	N %	H ppm
JK47	1.0016	1.070	0.0064	15.0
Iron Powder*	1.0035	1.093	0.0068	15.5
1.09% O	1.0098	1.088	0.0067	14.8
0.0067% N	1.0074	1.085	0.0067	14.6
LECO	1.0001	1.097	0.0068	14.7
502-399	1.0056	1.090	0.0067	14.3
	1.0040	1.098	0.0068	14.1
	1.0092	1.093	0.0067	15.0
	1.0018	1.086	0.0066	14.0
	1.0031	1.100	0.0067	14.7
	X =	***1.090***	***0.0067***	***14.7***
	s =	***0.009***	***0.0001***	***0.5***

**782-720S crucibles used.*

Delivering the Right Results

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