Hydrogen Determination in Steel and Iron

LECO Corporation; Saint Joseph, Michigan USA

Instrument: RHEN600/RHEN602

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

764-330[1] or 619-895[2] Graphite Crucibles, 761-739 Tin Pellets, 501-059 or 502-040 Tin Capsules.

[1]Use 783-568 Electrode Tip. [2]Use 619-896 Electrode Tip.

Calibration Samples

LECO 501-529, 762-747 One-Gram Steel Pins, NIST or other suitable reference materials.

Method Parameters

Analysis Parameters

Outgas Cycles	3
Analysis Delay	90 seconds
Analysis Delay Comparator	1.000
Analysis Type	Auto Analysis
Pre-Analysis Crucible Outgas	Disabled

Element Parameters	*Hydrogen*
Minimum Analysis Time	110 seconds
Significant Digits	7
Conversion Factor	1.000000
Integration Delay	30-40 seconds*
Comparator Level	2.000000%
Stop if Below (%)	0.000000

**Flow and column related; nominal is 35 seconds.*

Furnace Parameters

Furnace Control Mode	Current
Pre-Analyze Purge Time	—
Purge Time	15 seconds
Outgas Time	30 seconds
Outgas Cool Time	5 seconds
Outgas Low Power	900 amps**
Outgas High Power	900 amps**
Outgas Ramp Rate	—
Analyze Low Power	825 amps**
Analyze High Power	825 amps**
Analyze Ramp Rate	—
Sample Prep Time	—
Sample Prep Power	—
Furnace On Time	60 seconds
Temperature Sustain	None

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

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Determine Blank.
   a. Enter 1.0000 g mass into Sample Login (F3) using "Blank" as sample name.
   b. Press Loader Switch on the 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 one 761-739 Tin Pellet into a graphite crucible.
   e. Place crucible on electrode pedestal.
   f. Press Loader Switch; the lower electrode will close and the analysis sequence will start and end automatically.
   g. Repeat steps 2a through 2f a minimum of three times.
   h. Set the blank following the procedure outlined in the operator's instruction manual.
3. Calibrate/Drift Correct.
   a. Weigh ~1.0 g of a calibration sample; enter mass and sample identification into Sample Login (F3).
   b. Press Loader Switch on the 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 one 761-739 Tin Pellet into a graphite crucible.
   f. Place crucible on the 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 three times for each calibration/drift sample used.
   i. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.

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4. Analyze Samples.
   a. Weigh ~1.0 g sample; enter mass and sample identification into Sample Login (F3).
   b. Proceed as directed in steps 3b through 3g.

Typical Results—Solid Samples

Sample	*Weight (g)*	*H ppm*
LECO 501-529	1.0034	5.81
Steel Pin	1.0022	5.66
@ 5.7 ±0.4	1.0011	5.81
ppm H	1.0029	5.62
	1.0030	5.58
	1.0042	5.65
	1.0048	5.77
	1.0021	5.77
	1.0070	5.75
	1.0022	5.78
	***X* =**	***5.72***
	***s* =**	***0.084***

Alternate Procedure—Powder/Chip Samples

If powder or chip samples are to be analyzed, they cannot be placed directly in the loading head. Samples can be weighed into tin capsules and be analyzed in the Auto Analysis mode. There are issues related to blank, limited volume/sample weight, and the increased time and manipulation to weigh samples in a capsule. However, good results and precision are obtainable using this method. In addition, there are two methods of manually loading a sample available to the RHEN600/602 user.

Manual Analysis—The electrodes are opened after outgas and the sample is inserted into the crucible. This method will result in higher and more erratic hydrogen blanks and is generally not recommended.

Manual Top Load—After outgas, the loading head is opened and the sample is dropped into the crucible. For powder samples, a LECO 617-997 Funnel can be inserted through loading head into crucible and the sample is transferred to crucible via the funnel. This option will limit the outgassed crucible's exposure to the atmosphere, reducing blank variability, subsequently improving precision. Therefore, the Manual Top Load function of the RHEN600/602 can be used to obtain good and precise hydrogen results.

Tin Capsule Procedure

1. Determine Blank.
   a. Enter 1.0000 g mass into Sample Login (F3) using "Blank" as sample name.
   b. Press Loader Switch on the front of furnace, after a short delay the loading head slide block will open.
   c. Place an empty tin capsule into open port at top of loading head.

      Note: Use same part number and lot number of capsules that will be used for the analysis of samples, leave capsule open.
   d. Press Loader Switch, the lower electrode will open.
   e. Place one 761-739 Tin Pellet into a 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 1a through 1g a minimum of three times.
   i. Set the blank following the procedure outlined in the operator's instruction manual.
2. Calibrate/Drift Correct.
   a. Weigh ~1 gram of calibration sample into a tin capsule; and enter mass and sample identification into Sample Login (F3).
   b. Press Loader Switch on the front of furnace, after a short delay the loading head slide block will open.
   c. Place capsule into open port at top of loading head.
   d. Press Loader Switch; the lower electrode will open.
   e. Place one 761-739 Tin Pellet into a 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 three times for each calibration/drift sample used.
   i. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.
3. Analyze Samples.
   a. Weigh ~1 gram sample into a tin capsule and enter mass and sample identification into Sample Login (F3).
   b. Proceed as directed in steps 2b through 2g.

Typical Results Using Tin Capsule-Powder Samples

Sample	*Weight (g)*	*H ppm*
Steel Powder	0.9867	3.08
	1.0333	3.36
	1.0178	3.18
	1.0151	3.53
	1.0295	3.26
	1.0258	3.43
	1.0328	3.43
	1.0032	3.14
	0.9971	3.14
	1.0208	3.67
	***X* =**	***3.32***
	***s* =**	***0.196***

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.
2. Determine Blank.
   a. Enter 1.0000 g mass into Sample Login (F3) using "Blank" as sample name.
   b. Press Loader Switch on the front of furnace; after a short delay the lower electrode will open.
   c. Place one 761-739 Tin Pellet into a graphite crucible.
   d. Place crucible on electrode pedestal.
   e. Press Loader Switch; the lower electrode will close and the outgas sequence will start automatically.
   f. 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.
   g. Place the 617-997 Funnel into the open loading head.
   h. Remove the funnel and press the Loader Switch; the loading head slide block will close and the analysis sequence will start and end automatically.
   i. Repeat steps 2a through 2h a minimum of three times.
   j. 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; enter mass and sample identification into Sample Login (F3).
   b. Press Loader Switch on the front of furnace; after a short delay the lower electrode will open.
   c. Place one 761-739 Tin Pellet into a graphite crucible.
   d. Place crucible on electrode pedestal.
   e. Press Loader Switch; the lower electrode will close and the outgas sequence will start automatically.
   f. 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.
   g. 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.
   h. Remove funnel, press Loader Switch, and the loading head slide block will close and the analysis sequence will start and end automatically.
   i. Repeat steps 3a through 3h a minimum of three times for each calibration/drift sample used.
   j. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.
4. Analyze Samples.
   a. Weigh ~1.0 g of sample; enter mass and sample identification in Sample Login (F3).
   b. Proceed as directed in steps 3b through 3h.

Typical Results Manual Top Load Procedure—Powder Sample

Sample	*Weight (g)*	*H ppm*
Iron Powder	1.0131	4.64
	1.0047	4.32
	1.0142	4.54
	1.0141	4.32
	1.0158	4.29
	1.0008	4.47
	1.0368	4.61
	1.0076	4.31
	1.0245	4.70
	1.0025	4.69
	***X* =**	***4.49***
	***s* =**	***0.169***

Inorganic Application Note

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