

Inorganic Application Note

Hydrogen Determination in Steel and Iron

Instrument

RH600

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

782-720 Graphite Crucibles, 761-739 Tin Pellets, 501-059 or 502-040 Tin Capsules

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

¹In 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.

Element Parameters

	Hydrogen
Minimum Analysis Time	60 seconds
Significant Digits	6
Conversion Factor	1.000000
Integration Delay	10 seconds
Comparator Level	1.000000 %
Stop if below (%)	0.000000

RH600

Furnace Parameters

Furnace Control Mode	Current
Pre-Analyze Purge Time	—
Purge Time	10 seconds
Outgas Time	15 seconds
Outgas Cool Time	5 seconds
Outgas Low Power	850 amps*
Outgas High Power	850 amps*
Outgas Ramp Rate	—
Analyze Low Power	765 amps*
Analyze High Power	765 amps *
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 g 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 one 761-739 Tin Pellet into a 782-720 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 five 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 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 one 761-739 Tin Pellet into a 782-720 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 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 g sample and enter weight into weight stack.
 - b. Proceed as directed in steps 3b through 3f.

Typical Results—Solid Samples

Sample	Weight g	H ppm
LECO	1.0003	5.45
501-529	1.0020	5.74
Steel Pin	1.0025	5.59
@5.7 ±0.4	1.0021	5.56
ppm H	1.0083	5.67
	1.0009	5.64
	1.0045	5.24
	1.0026	5.88
	0.9972	5.40
	1.0079	5.79
	X=	5.60
	s =	0.19

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 Semi-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 RH600 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, 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 limits the outgassed crucible exposure to the atmosphere, reducing blank variability, subsequently improving precision. Therefore, the Manual Top Load function of the RH600 can be used to obtain good and precise hydrogen results.

Tin Capsule Procedure

- Determine Blank.
 - Enter 1.0000 g weight into weight stack.
 - Press Loader Switch on front of furnace, after a short delay the loading head slide block will open.
 - 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.
 - Press Loader Switch, the lower electrode will open.
 - Place one 761-739 Tin Pellet into a 782-720 Graphite Crucible.
 - Place crucible on electrode pedestal.
 - Press Loader Switch, the lower electrode will close and the analysis sequence will start and end automatically.
 - Repeat steps 1a through 1g a minimum of five times.
 - Set the blank following the procedure outlined in the operator's instruction manual.
- Calibrate/Drift Correct.
 - Weigh ~1 gram of calibration sample into a tin capsule and enter weight into weight stack.
 - Press Loader Switch on front of furnace, after a short delay the loading head slide block will open.
 - Place capsule into open port at top of loading head.
 - Press Loader Switch, the lower electrode will open.
 - Place one 761-739 Tin Pellet into a 782-720 Graphite Crucible.
 - Place crucible on electrode pedestal.
 - 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 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.
3. Analyze Samples.
- a. Weigh ~1 gram sample into a tin capsule and enter weight into weight stack.
 - b. Proceed as directed in steps 2b through 2g.

Typical Results—Powder Samples

Sample	Weight	H ppm
Steel Powder	1.0073	10.84
	1.0063	10.89
	1.0020	10.88
	1.0086	11.04
	0.9993	10.69
	1.0079	10.60
	1.0059	10.62
	1.0088	10.69
	1.0086	11.13
	1.0061	11.23
	X =	10.86
	s =	0.22

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 weight into weight stack.
 - b. Press Loader Switch on front of furnace, after a short delay the lower electrode will open.
 - c. Place one 761-739 Tin Pellet into a 782-720 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 five 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 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 one 761-739 Tin Pellet into a 782-720 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, 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 five 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 and enter weight into weight stack.
 - b. Proceed as directed in steps 3b through 3h.

Typical Results Manual Top Load Procedure—Powder Sample

Sample	Weight	H ppm	
JK47	1.0016	14.98	
Iron Powder	1.0035	15.50	
	1.0098	14.80	
	1.0074	14.55	
	1.0001	14.68	
	1.0056	14.26	
	1.0040	14.10	
	1.0092	14.95	
	1.0018	13.99	
	1.0031	14.67	
	X =	14.65	
	s =	0.452	



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