Application Note

Instrument: H836EN

Determination of Hydrogen in Steel and Iron

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Introduction

Hydrogen determination in steel and iron is one of the most important quality metrics for these materials. High Hydrogen content is the primary cause of embrittlement, blistering, and flaking due to its high mobility through the lattice, and provides no potential alloying benefits. Subsequently, the determination of Hydrogen in steel and iron is a critical quality control process.

Instrument Model and Configuration

The H836EN instrument is optimized for low level Hydrogen determination in steel, iron, refractory metals, and other inorganic materials. A pre-weighed sample is placed in a graphite crucible, which is heated in an impulse furnace to release analyte gases into a flowing stream of Argon carrier gas. Evolved Oxygen reacts with the graphite crucible to form CO and CO_2. Separate reagents remove moisture from the analyte gases present as H_2O and convert CO to CO_2 which is then scrubbed from the analyte gases by an additional reagent. A molecular sieve column is used to separate the analyte gases H_2 and N_2. Then, a thermal conductivity (TC) cell is used for the detection of Hydrogen (H_2).

Sample Preparation

Proper sampling and sample preparation techniques for Hydrogen determination in steel and iron are crucial for optimizing the accuracy and precision of the results. It is important to understand that traditional methods used to obtain samples for Oxygen and Nitrogen determination are different from those recommended for Hydrogen determination, especially when sampling molten metal. The main difference between steel and iron sampling procedures for Oxygen and Nitrogen, compared to that for 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 the loss of Hydrogen from diffusion. Losses of Oxygen and Nitrogen from diffusion are not a problem. A sample that is typically taken for Oxygen and/or Nitrogen determination is not suitable for Hydrogen determination due to Hydrogen loss (from diffusion).

Surface contamination must be removed from solid samples by filing or light grinding, using care not to overheat the sample. Subsequently, the prepared sample is rinsed using a suitable solvent, such as reagent grade acetone and then dried with warm air. The prepared sample must be handled with clean tweezers and analyzed immediately following preparation. Chip and powder samples should be a uniform mesh size. ASTM E1806 and ISO 14284 are sampling/sample preparation methods specific for steel and iron and are an excellent source of information.

Note: LECO Reference Materials typically do not require preparation prior to analysis. Refer to preparation statement on the reference material certificate.

Accessories

619-895 High Temperature Graphite Crucibles, 761-739 Tin Flux Pellets, 502-040 Tin Capsules, 619-896 Lower Electrode Tip, 766-053 Crucible Tweezers, 760-138 Sample Tweezers.

Reference Materials

LCRM®, LRM®, NIST, or other suitable reference materials.

*Method Parameters**

General Parameters	
Sample Introduction	Automated Sample Drop
Analysis Delay	70 s
Wait For User To Load Sample	Yes
Auto Analyzer on Mass Entry	No
Outgas Before Mass Entry	No
Outgas At End Of Analysis	No
Vacuum On Time	20 s
Element Parameters	
Integration Delay	30 s
Starting Baseline	2 s
Use Comparator	Yes
Comparator Level	0.10 %
Integrate from Furnace Method	No
Minimum Integration Time	70 s
Maximum Integration Time	110 s
Furnace Parameters**	
Furnace Control Mode	Current
Outgas Parameters	
Cycles	3
Current Mode	Constant
Current	900 A†
Time	30 s
Cool Time	5 s

Analyze Furnace Settings

Step 1

Current Mode	Constant
Current	850 A[†]
Time	60 s

Step 2

Current Mode	Constant
Current	0 A
Time	30 s

**Refer to 836 Series Operator's Instruction Manual for parameter definitions.*

***Select an appropriate existing furnace method and edit parameter settings as needed, or create a new furnace method with the appropriate parameter settings.*

[†]May vary based on the line voltage. Adjust to improve recovery or to reduce crucible burn-through.

Note: For Hydrogen detection below 1 ppm, bypassing the OMI Scrubber may improve precision. Refer to the 836 Series Operator's Instruction Manual for instructions on how to bypass the OMI Scrubber.

Procedure - Solid Samples

1. Prepare the instrument as outlined in the operator's instruction manual.
2. Determine the instrument blank.
   a. Log in a minimum of three Blank replicates and select the appropriate Method. Then select the appropriate Furnace Method.
   b. Press the Analyze button on the instrument screen. After a short delay, the loading head slide-block will open.
   c. Press the Analyze button on the instrument screen again. The loading head slide-block will close, and the lower electrode will open.
   d. Clean the upper and lower electrode either manually or with an equipped automatic cleaner.
   e. Add 1 Pellet of 761-739 Tin Flux to a 619-895 Graphite Crucible.
   f. Firmly place the graphite crucible containing the tin flux on the lower electrode tip.
   g. Press the Analyze button on the instrument screen and the lower electrode will close. The analysis sequence will start and end automatically.
   h. Perform steps 2b through 2g a minimum of three times.
   i. Set the blank following the procedure outlined in the operator's instruction manual.
3. Instrument calibration/drift correction.
   a. Log in a minimum of three Standard replicates and select the appropriate Method. Then select the appropriate Furnace Method.
   b. Weigh ~1.0 g of a suitable calibration/drift sample and enter the mass and sample identification into the appropriate replicate fields.
   c. Press the Analyze button on the instrument screen. After a short delay, the loading head slide-block will open.
   d. Place the calibration/drift sample into the open port at the top of the loading head.
   e. Press the Analyze button on the instrument screen again. The loading head slide-block will close, and the lower electrode will open.
   f. Clean the upper and lower electrode either manually or with an equipped automatic cleaner.
   g. Add 1 Pellet of 761-739 Tin Flux to a 619-895 Graphite Crucible.
   h. Firmly place the graphite crucible containing the tin flux on the lower electrode tip.
   i. Press the Analyze button on the instrument screen and the lower electrode will close. The analysis sequence will start and end automatically.
   j. Perform steps 3b through 3i a minimum of three times for each calibration/drift sample utilized.
   k. Calibrate/drift following the procedure outlined in the operator's instruction manual.
4. Analyze Samples.
   a. Log in a Sample with the appropriate number of replicates and select the appropriate Method. Then select the appropriate Furnace Method.
   b. Weigh ~1.0 g of a prepared sample and enter the mass and sample identification into the appropriate replicate fields.
   c. Press the Analyze button on the instrument screen. After a short delay, the loading head slide-block will open.
   d. Place the sample into the open port at the top of the loading head.
   e. Press the Analyze button on the instrument screen again. The loading head slide-block will close, and the lower electrode will open.
   f. Clean the upper and lower electrode either manually or with an equipped automatic cleaner.
   g. Add 1 Pellet of 761-739 Tin Flux to a 619-895 Graphite Crucible.
   h. Firmly place the graphite crucible containing the tin flux on the lower electrode tip.
   i. Press the Analyze button on the instrument screen and the lower electrode will close. The analysis sequence will start and end automatically.
   j. Perform steps 4b through 4i for each sample being analyzed.

Procedure – Chip/Powder Samples

Note: Chip and powder samples should be a uniform mesh size. Chip samples with particle sizes of +40 mesh (420 microns) may be analyzed following the procedure outlined for analysis of solid samples. Chip samples with particles sizes of -40 mesh must be analyzed following the procedure outlined below.

1. Prepare the instrument as outlined in the operator's instruction manual.
2. Determine the instrument blank.
   a. Log in a minimum of three Blank replicates and select the appropriate Method. Then select the appropriate Furnace Method.
   b. Press the Analyze button on the instrument screen. After a short delay, the loading head slide-block will open.
   c. Place a 502-040 Tin Capsule (leave capsule open) into the open port at the top of the loading head.
   d. Press the Analyze button on the instrument screen again. The loading head slide-block will close, and the lower electrode will open.
   e. Clean the upper and lower electrode either manually or with an equipped automatic cleaner.
   f. Add 1 Pellet of 761-739 Tin Flux to a 619-895 Graphite Crucible.

g. Firmly place the graphite crucible containing the tin flux on the lower electrode tip.
h. Press the Analyze button on the instrument screen and the lower electrode will close. The analysis sequence will start and end automatically.
i. Perform steps 2b through 2h a minimum of three times.
j. Set the blank following the procedure outlined in the operator's instruction manual.

3. Instrument calibration/drift correction.
   a. Log in a minimum of three Standard replicates and select the appropriate Method. Then select the appropriate Furnace Method.
   b. Weigh ~1.0 g of a suitable calibration/drift sample into a 502-040 Tin Capsule and enter the mass and sample identification into the appropriate replicate fields.

   Note: Solid reference materials may be utilized to calibrate when chip or powder reference materials are not available.

   c. Press the Analyze button on the instrument screen. After a short delay the loading head slide-block will open.
   d. Place the tin capsule containing the calibration/drift sample into the open port at the top of the loading head.
   e. Press the Analyze button on the instrument screen again. The loading head slide-block will close, and the lower electrode will open.
   f. Clean the upper and lower electrode either manually or with an equipped automatic cleaner.
   g. Add 1 Pellet of 761-739 Tin Flux to a 619-895 Graphite Crucible.
   h. Firmly place the graphite crucible containing the tin flux on the lower electrode tip.
   i. Press the Analyze button on the instrument screen and the lower electrode will close. The analysis sequence will start and end automatically.
   j. Perform steps 3b through 3i a minimum of three times for each calibration/drift sample utilized.
   k. Calibrate/drift following the procedure outlined in the operator's instruction manual.

4. Analyze Samples.
   a. Log in a Sample with the appropriate number of replicates and select the appropriate Method. Then select the appropriate Furnace Method.
   b. Weigh ~1.0 g of a sample into a 502-040 Tin Capsule and enter the mass and sample identification into the appropriate replicate fields.
   c. Press the Analyze button on the instrument screen. After a short delay the loading head slide-block will open.
   d. Place the tin capsule containing the sample into the open port at the top of the loading head.
   e. Press the Analyze button on the instrument screen again. The loading head slide-block will close, and the lower electrode will open.
   f. Clean the upper and lower electrode either manually or with an equipped automatic cleaner.
   g. Add 1 Pellet of 761-739 Tin Flux to a 619-895 Graphite Crucible.
   h. Firmly place the graphite crucible containing the tin flux on the lower electrode tip.
   i. Press the Analyze button on the instrument screen and the lower electrode will close. The analysis sequence will start and end automatically.
   j. Perform steps 4b through 4i for each sample being analyzed.

Typical Results

Results are based on a linear, force through origin calibration utilizing 502-963 (Lot: 0737) LCRM Steel Pins (6.7 ppm Hydrogen).

Solid Sample

Sample	*Mass (g)*	*Hydrogen (ppm)*
502-913	0.9932	3.27
Steel Pin	1.0025	3.08
3.5 ±0.6 ppm H	0.9921	3.37
	1.0021	3.23
	1.0027	3.12
	1.0037	3.22
	1.0049	3.52
	1.0016	3.14
	1.0036	3.19
	1.0030	3.39
	Avg =	***3.25***
	s =	***0.14***

Chip/Powder Samples

Sample	*Mass (g)*	*Hydrogen (ppm)*
Steel Chip	1.0160	2.76
	1.0067	2.73
	1.0115	2.61
	0.9996	2.76
	0.9912	2.61
	0.9974	2.89
	1.0082	2.74
	1.0112	2.91
	0.9972	2.73
	0.9955	2.92
	Avg =	***2.77***
	s =	***0.11***
Iron Powder	0.9794	3.33
	0.9909	3.54
	0.9956	3.19
	1.0089	2.95
	1.0046	3.12
	1.0023	3.27
	1.0014	3.24
	1.0070	3.36
	1.0024	3.06
	0.9953	3.10
	Avg =	***3.21***
	s =	***0.17***

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