

Hydrogen in Steel Samples

LECO Corporation; Saint Joseph, Michigan USA

Instrument: DH603

Introduction

The determination of hydrogen in steel is a time-tested quality control parameter. Excess amounts of hydrogen present during the solidification and cooling process has an adverse effect on both physical and mechanical parameters including embrittlement, porosity, cracking, and flaking.

This application poses a unique challenge due to the rapid diffusion of hydrogen from steel during sampling. To accommodate this, a vacuum pin tube sampler is employed that traps the diffusible hydrogen in an outer enclosure and retains the solidified sample (pin) for residual hydrogen analysis and correlation.

The DH603 allows the analyst to determine the amount of hydrogen present in its molten form and after solidification and cooling by incorporating an optional vacuum pin tube sample piercer for diffusible hydrogen determination and a hot extraction furnace for residual hydrogen determination.

Accessories Tweezers

Sample Size Nominal 5 g

Furnace Temp 500°C standby and 1100°C Analyze

Calibration Samples

LECO 502-060, 502-061, or other suitable reference materials.

NOTE: To burn-off push rod, enter 5.0000 g mass into Sample Login (F3) using Burn Off as the sample name. Place the rod in the furnace at 1100°C for 90 seconds. Remove the rod and let cool for at least 5 minutes before running a residual analysis. Repeat burn-off procedure every 8 hours or as needed. Between analyses place rod so that tip of rod is not touching anything.

Element Parameters

	Diffusible H	Residual H
Baseline Time (seconds)	2	2
Endline Time (seconds)	1	1
Significant Digits	7	7
Conversion Factor	1.00	1.00
Analysis Stabilize Comparator	1.00	1.00
Integration Delay (seconds)	5	5

Gas Dose Parameters

	Hydrogen
Baseline Time (seconds)	2
Endline Time (seconds)	1
Significant Digits	7
Conversion Factor	1.00
Analysis Stabilize Comparator	1.00
Integration Delay	15



System Parameters

Conservation Timeout (minutes)	180
Pre-Analysis Purge Time (seconds)	10
Nominal Mass for Blanks	5 g
Auto Increment Sample Name	Disable
Standby Flow	0.12 lpm
Furnace Standby Temperature	500°C
Ambient Temperature	Disable
Furnace Start Up Temperature	1000°C
Oven Temperature	50
Enable Piercer Unit	Enable
<i>(requires software restart when changed)</i>	
Diffusible Purge Time (seconds)	15

Furnace Step Method

Gas Dose Hold	Gas Dose			
Diffusible Hydrogen Temperature	1100°C			
Diffusible Hydrogen Hold	Diffusible			
Residual Hydrogen Furnace Steps				
Name	Target	Ramp	Hold	Estimated Time
Start	1100	N/A	Residual	300-1000

Hold Parameters

Gas Dose	Hydrogen
Minimum Analysis Time (seconds)	65
Maximum Analysis Time (seconds)	130
Comparator Level	1.00
Diffusible	Hydrogen
Minimum Analysis Time (seconds)	150
Maximum Analysis Time (seconds)	200
Comparator Level	2.00
Residual	Hydrogen
Minimum Analysis Time (seconds)	300
Maximum Analysis Time (seconds)	1000
Comparator Level	2.00

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Determine Sample Blank.
 - a. Enter 5.0 g mass into Sample Login (F3) using Blank as the sample name, select Residual as type of analysis.
 - b. Start analysis by initiating Analyze (F5) (Reaction tube door closed).
 - c. When User Loading Sample message appears in the lower left part of the screen and Load Sample display box appears on center of screen, click OK or press the enter key. Open reaction tube door and slide push rod into tube until it reaches the boat stop; remove push rod, close door.

- d. When analysis is complete, repeat steps 2a through 2c a minimum of three times.
- e. Set the blank following the procedure outlined in the operator's instruction manual.
3. Calibrate/Drift Correct.
 - a. Weigh ~5.0 g of the 502-060 calibration sample; enter mass and sample identification into Sample Login (F3), select Residual as type of analysis.
 - b. Start analysis by initiating Analyze (F5) (Reaction tube door closed).
 - c. When User Loading Sample message appears in the lower left part of the screen and Load Sample display box appears on center of screen, click OK or press the enter key. Open reaction tube door and load sample into furnace, using push rod to slide sample into tube until it reaches the boat stop; remove push rod, close door.
 - d. When analysis is complete, remove sample using the push rod and close door.
 - e. Repeat steps 3a through 3d a minimum of three times.
 - f. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual (Single Standard Calibration Recommended).
4. Analyze Diffusible/Residual Samples.
 - a. Enter 5.0 g mass and sample identification into Sample Login (F3) as the sample name, select Both as type of analysis.
 - b. Push the steel sampler tube firmly into the piercer unit and hold it until the piercer seals clamp the tube.
- c. The diffusible analysis will begin automatically when the piercing unit senses a sample. The hydrogen concentration will be displayed in the diffusible window.
- d. When finished, the piercing unit will release the sampler.
- e. Extract the pin sample from the metal sampler tube by cutting the outer tubing 3/8 inch from the collar with a tube cutter or other suitable device. Remove and discard the outer tube.
- f. Cut the pin as close to the base collar as possible using a hacksaw or bolt cutter. Avoid contaminating the sample; the device used for cutting should be free from foreign material such as paint. Do not heat the sample while cutting; this can drive hydrogen from the sample.
- g. When User Loading Sample message appears in the lower left part of the screen and Load Sample display box appears on center of screen, click OK or press the Enter key. Open reaction tube door and load sample into furnace, using push rod to slide sample into tube until it reaches the boat stop; remove push rod, close door.
- h. When analysis is complete, remove sample using the push rod and close door.
5. Analyze Residual (only) Samples.
 - a. Weigh ~5.0 g of the sample; enter mass and sample identification into Sample Login (F3), select Residual type of analysis.
 - b. Proceed as directed in steps 3b through 3d.

Typical Results

Sample	Mass g	Diffusible H ppm	Residual H ppm	Total H ppm
502-060	4.9974		5.87	5.87
@ 6.0 ppm H	4.9796		5.73	5.73
±0.3	4.9981		6.01	6.01
	5.0034		6.14	6.14
	4.9960		6.04	6.04
		X =	5.96	5.96
		s =	0.16	0.16
502-061	4.9925		2.15	2.15
@ 2.19 ppm H	5.0062		2.08	2.08
±0.35	4.9891		2.14	2.14
	4.9966		2.20	2.20
	5.0269		2.18	2.18
		X =	2.15	2.15
		s =	0.05	0.05

Sample	Mass g	Diffusible H ppm	Residual H ppm	Total H ppm
Steel Sampler	5.000	7.31	3.05	10.36
LECO 903-9A3-184	5.000	7.79	3.13	10.92



LECO Corporation

3000 Lakeview Avenue • St. Joseph, MI 49085 • Phone: 800-292-6141 • Fax: 269-982-8977
 info@leco.com • www.leco.com • ISO-9001:2000 • No. FM 24045 • LECO is a registered trademark of LECO Corporation.