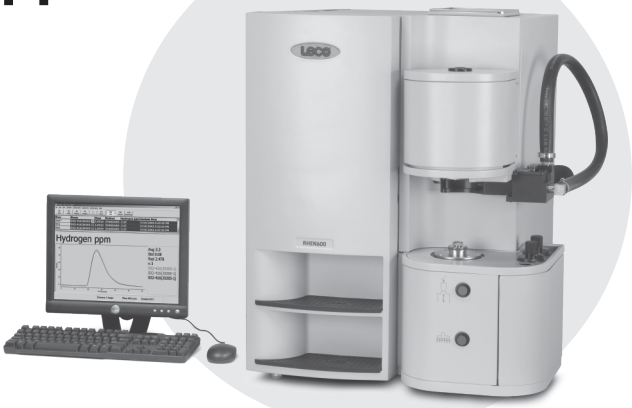
Inorganic Application Note

Hydrogen Determination in Copper and Copper Alloys



Instrument

RHEN600/602

Introduction

Copper is an integral component in the electronics and household product industries due to its advantageous electrical and thermal properties. Copper wires or copper-containing components can be found in virtually all electronic components, while copper plumbing and copper-based flatware/cookware are most obvious in the home. Copper is also readily found in coinage, biomedical applications, chemical applications, and musical instruments.

Due to the sensitivity of the aforementioned copper products, an emphasis on purity must be maintained by the copper producers. One impurity of particular interest is hydrogen. Hydrogen is readily absorbed in molten copper during processing, but is readily liberated during the solidification process. The presence of occluded hydrogen indicates the potential for porosity, diminished ductility, and reduced thermal and electrical conductivity. Thus, an accurate hydrogen determinator is a necessary quality control device for all copper producers.

The LECO RHEN600 and RHEN602 are hydrogen determinators that utilize an electrode furnace, an argon carrier gas, and thermal conductivity detection to meet the needs of the metals, refractory, and inorganic material industries. The RHEN602 has the added ability to differentiate between surface and bulk hydrogen when analyzing aluminum and aluminum alloys.

This application note was written for use with the LECO RHEN600 or RHEN602 Hydrogen Determinator.

Sampling and Sample Preparation

Samples must be representative and free of surface contamination. Solid samples can be abraded or milled to remove surface contamination.

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 782-721 or 618-376 Electrode Tip

Calibration Samples

LECO 501-529, 762-747 One-Gram Steel Pins, NIST or other suitable reference materials. Gas dosing may also be used to calibrate/drift correct, refer to the operator's instruction manual.

RHEN600/602

Method Parameters

Analysis Parameters	
Outgas Cycles	3
Analysis Delay	90 seconds
Analysis Delay Comparator	1.000
Analysis Type	Semi-Auto Analysis
Pre-Analysis Crucible Outgas	Disabled
Bulk/Surface Separation (RHEN602 only)	Disabled

Element Parameters	*Hydrogen*
Minimum Analysis Time	70 seconds
Significant Digits	7
Conversion Factor	1.000000
Integration Delay	40 seconds
Comparator Level	2.000000 %
Stop if Below (%)	0.000000

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	—
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 and "Blank" as sample identification into Sample Login (F3).
   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 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 to 3.0 g of a calibration sample, enter mass and sample identification information into Sample Login (F3).
   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 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.

Note: Gas dosing may be used to calibrate/drift correct in lieu of analyzing solid calibration samples. Refer to the operator's instruction manual for details.

4. Analyze Samples.
   a. Weigh ~1.0 to 3.0 g sample, enter mass and sample identification information into Sample Login (F3).
   b. Proceed as directed in steps 3b through 3f.

Typical Results—Solid Samples

Sample	Mass g	H ppm*
Oxygen Free	1.6641	1.43
Copper Slug	1.7694	1.40
(0.001% O	1.7732	1.30
max)	1.8587	1.43
	1.7922	1.39
	X =	***1.39***
	s =	***0.053***
OFE	2.0074	0.38
Copper Rod	1.8594	0.33
(0.0005% O	1.9028	0.36
max)	1.8375	0.32
	1.8730	0.31
	X =	***0.34***
	s =	***0.031***

**Results based on a single standard calibration with LECO 501-529 Steel Pin Sample.*

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.

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.

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

NOTE: The manual methods will result in higher and more erratic hydrogen blanks, therefore they are not recommended for this application.

Tin Capsule Procedure

1. Determine Blank.
   a. Enter 1.0000 g mass and "Blank" as sample identification into sample login (F3)
   b. Press Loader Switch on 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 five times.
i. Set the blank following the procedure outlined in the operator's instruction manual.

2. Calibrate/Drift Correct.
   a. Weigh ~1.0 g of calibration sample into a tin capsule and enter mass and sample identification into Sample Login (F3).

   Note: Calibration/Drift samples can be solid; they do not have to be powder or chip. Gas dosing may be used in lieu of analyzing calibration samples. Refer to operator's instruction manual for details.

   b. Press Loader Switch on 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 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 g sample into a tin capsule and enter mass and sample identification into Sample Login (F3).
   b. Proceed as directed in steps 2b through 2g.

   Note: Sample mass can be reduced if sample contains higher levels of hydrogen or the material "bulk" will not permit larger sample masses to fit into the capsule.

Typical Results—Tin Capsule-Powder Samples

Sample	Mass g	H ppm*
Copper Powder	0.8152	124.6
Unknown	0.8245	124.9
(-300 mesh)	0.8916	124.9
	0.9201	123.2
	0.9670	124.0
	X =	***124.3***
	s =	***0.70***

**Results based on a single standard calibration with LECO 502-339 Hydrogen in Titanium Sample, and checked with LECO 501-529 Hydrogen in Steel Calibration Sample.*

Delivering the Right Results

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