# Application Note

## *Instrument: RHEN602*

# Hydrogen Determination in Aluminum and Aluminum Alloys

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## *Introduction*[1, 2]

Aluminum has several qualities that make it the preferred material for applications in the construction, aerospace, electronics, container, and automotive industries. These qualities include low density, high strength, high conductivity, a desirable appearance, and superior corrosion resistance. Unfortunately, aluminum also has some disadvantages, including a relatively high cost and unfavorable hydrogen solubility characteristics.

Hydrogen gas has a high solubility in molten aluminum and is readily introduced during processing. Once solidification begins, the solubility decreases approximately twenty fold, and hydrogen is forced out of the aluminum. Although a majority of the hydrogen is diffused, a small percentage may get trapped, creating hydrogen filled voids. This remaining hydrogen gas is the root cause of numerous failure mechanisms in aluminum products such as voids in casting and blisters in sheets. Thus, an accurate hydrogen determinator is a necessary quality control device for all aluminum producers.

The LECO RHEN602 is a hydrogen determinator which utilizes an electrode furnace, an argon carrier gas, and thermal conductivity detection to meet the needs of the aluminum industry. The RHEN602 software takes advantage of the existing ability to perform stepped furnace analysis and incorporates it in such a way that separation of surface and bulk hydrogen can be performed and corresponding results reported.

Each of these steps serves a specific purpose for this analysis.

- Step 1 is used to liberate surface hydrogen
- Step 2 provides the surface hydrogen time to separate from the bulk hydrogen
- Step 3 is optional and used to ensure complete liberation of surface hydrogen prior to bulk analysis
- Step 4 is used to liberate bulk hydrogen
- Step 5 is a power reduction step used to prevent aluminum from boiling into the upper electrode

The RHEN602 software allows for separate programming of minimum analysis time and analysis delay for surface and bulk hydrogen. The acquisition screen plots two different windows (Surface and Bulk) based on these values. Next, the Surface and Bulk values can be summed and the Total Hydrogen value reported.

## *Sampling and Sample Preparation*

This application is written for solid aluminum samples. Samples are sectioned, and machined on a lathe to a uniform sample dimension. This facilitates surface/bulk hydrogen determination. Sampling of molten metal is typically done using a well-fed chill wedge-bar copper book mold (referred to as a Ransley mold[3]) to minimize formation of voids. The wedge is separated from the bar using a water-cooled cut-off saw. Wrought products are sectioned using a water-cooled cut-off saw. Final sample preparation is completed using a lathe. Lathe chuck and tool must be clean and free of lubricants. Nominal lathe speed is 600 rpm to 700 rpm, advance at ~0.04 mm/revolution. It is important that the entire original surface is removed. The maximum/ideal sample dimension is 10.5 x 24 mm. The sample may be rinsed in acetone and dried with warm air prior to analysis to ensure the removal of any remaining surface contamination. Prepared sample should be analyzed as quickly as possible to minimize surface contamination/hydrogen pick-up. Handle prepared sample with clean tweezers.

*Click here to watch an Aluminum Sample Preparation video.*

## *Accessories*

764-330 Graphite Crucibles, 611-351-183 Electrode Tip.

## *Reference Materials*

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

## *Method Parameters*

*Analysis Parameters*

| | |
|---|---|
| Outgas Cycles | 4 |
| Analysis Delay | 120 s |
| Analysis Delay Comparator | 1.000 |
| Analysis Type | Semi-Auto Analysis |
| Pre-Analysis Crucible Outgas | Disabled |

| *Element Parameters* | *Hydrogen Surface* | *Hydrogen Bulk* | *Standards** |
|---|---|---|---|
| Minimum Analysis Time | 130 s | 340 s | 90 s |
| Significant Digits | 7 | 7 | 7 |
| Conversion Factor | 1.000000 | 1.000000 | 1.000000 |
| Integration Delay | 35 s | 155 s | 32 s |
| Comparator Level | 2.000000% | 2.000000% | 2.000000% |

**When steel calibration samples are used for calibration.*

| *Gas Dose Parameters** | *Hydrogen* |
|---|---|
| Minimum Gas Dose Time | 70 s |
| Integration Delay | 32 s |
| Comparator Level | 1.000000% |
| Bypass Furnace | Yes (check box) |
| Gas Dose Cycles | 1 |

**Helium or Nitrogen may also be used as dose gases.*

*Furnace ParametersStandards**

| Furnace Control Mode | Power | Power |
|---|---|---|
| Purge Time | 15 s | 15 s |
| Outgas Time | 30 s | 15 s |
| Outgas Cool Time | 5 s | 5 s |
| Outgas Low Power | 4500 W | 4500 W |
| Outgas High Power | 4500 W | 4500 W |
| Outgas Ramp Rate | 0 W/s | 0 W/s |
| Analyze Low Power | 0 W | 3700 W |
| Analyze High Power | 1400 W | 3700 W |
| Analyze Ramp Rate | 0 W/s | 0 W/s |
| Sample Prep Time | 0 s | 0 s |
| Sample Prep Power | 0 W | 0 W |
| Furnace On Time | 300 s | 70 s |

**When steel or titanium calibration sample are used for calibration.*

*Temperature Sustain*

| *Step* | *Start Power* | *End Power* | *Time* |
|---|---|---|---|
| 1 | 900 W | 900 W | 20 s |
| 2 | 0 W | 0 W | 60 s |
| 3 | 450 W | 450 W | 70 s |
| 4 | 1400 W | 1400 W | 30 s |
| 5 | 0 W | 0 W | 150 s |

Temperature Sustain Standards: None

## *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.
   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 crucible on electrode pedestal.
   e. Press Loader Switch, the lower electrode will close and the analysis sequence will start and end automatically.
   f. Repeat steps 2a through 2e a minimum of five times.
   g. Set the blank following the procedure outlined in the operator's instruction manual.
3. Calibrate/Drift Correct.
   a. Weigh a calibration sample and enter mass into sample login.
   b. Press Loader Switch on front of furnace, the loading head slide block will open.
   c. Place prepared 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 crucible on the electrode pedestal.
   f. Press Loader Switch, the lower electrode will close and the analysis sequence will start and end automatically.
   g. Repeat steps 3a through 3f a minimum of five times for each calibration/drift sample used.
   h. 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 ~3.0 to 6.5 g prepared aluminum sample and enter mass into sample login.
   b. Proceed as directed in steps 3b through 3f.

## *Typical Results—Solid Samples**

| *Sample* | *Mass g* | *Surface H ppm* | *Bulk H ppm* | *Total H ppm* |
|---|---|---|---|---|
| 6061 | 5.8061 | 0.017 | 0.065 | 0.082 |
| Aluminum Alloy | 5.9035 | 0.026 | 0.057 | 0.083 |
| | 5.3544 | 0.022 | 0.058 | 0.080 |
| | 5.1521 | 0.026 | 0.061 | 0.087 |
| | ***Avg =*** | ***0.023*** | ***0.060*** | ***0.083*** |
| | ***s =*** | ***0.0043*** | ***0.0033*** | ***0.0029*** |
| AlMg 5% | 3.8328 | 0.050 | 0.150 | 0.200 |
| Aluminum Alloy | 3.9159 | 0.047 | 0.174 | 0.221 |
| 0.16 ppm H (bulk) | 3.4060 | 0.059 | 0.160 | 0.219 |
| Reference Standard** | 3.9018 | 0.049 | 0.166 | 0.215 |
| | ***Avg =*** | ***0.051*** | ***0.163*** | ***0.214*** |
| | ***s =*** | ***0.0054*** | ***0.0101*** | ***0.0095*** |
| AlMgSi 0.5% | 6.3068 | 0.026 | 0.133 | 0.159 |
| Aluminum Alloy | 6.1883 | 0.037 | 0.128 | 0.165 |
| 0.14 ppm H (bulk) | 6.0590 | 0.034 | 0.140 | 0.174 |
| Reference Standard** | 5.7480 | 0.020 | 0.138 | 0.158 |
| | ***Avg =*** | ***0.029*** | ***0.135*** | ***0.164*** |
| | ***s =*** | ***0.0080*** | ***0.0051*** | ***0.0073*** |

**Results based on calibration with LECO 501-529 Steel Pin.*
***Al-reference materials from Senter for Industriforskning, Oslo, Norway.*

## *References*


[1]D. E.J. Talbot, "The Effects of Hydrogen in Aluminum and Its Alloys", Maney, London, 2004.

[2]P. D. Hess and G. K. Turnbull, Proceedings of International Conference on the Effects of Hydrogen on Materials Properties and Selection and Structural Design, Champion, 1973, 277-287.

[3]C.E. Ransley and D.E.J. Talbot, "The Routine Determination of the Hydrogen Content of Aluminum and Aluminum Alloys by the Hot-Extraction Method". Journal of the Institute of Metals, Vol. 84, 1955-1956, 445.




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