Application Note

Instrument: RC612

Determination of Moisture in Welding Flux

LECO Corporation; Saint Joseph, Michigan USA

Introduction

Weld flux provides many benefits during the welding process. The flux material can help with the initiation of the welding arc, especially when welding in AC. The flux will decompose under the heat of the weld and produce fumes that protect the raw weld from atmospheric exposure, preventing potential oxidation. The decomposed flux will become slag that floats to the surface of the weld pool protecting the surface of the weld and slowing the cooling process. Rapid cooling is known to promote detrimental microstructural changes in the weld metal. Moisture content in the flux can be correlated to the hydrogen content in the weld metal, and to varying degrees, the potential for hydrogen embrittlement. The potential weld quality can then be estimated based on the moisture content of the weld flux or other weld consumables. The LECO RC612 can quantify the amount of moisture present in welding flux by infrared measurement of the moisture evolved from a heated sample. The following application note outlines the parameters and procedures for moisture determination in weld flux with the RC612.

Accessories

781-335 Quartz Boat; 782-059 Nickel Liners; 625-505-430 Nickel Boat and 502-156 Fluorhib (preheated)*; Capillary Tube (sealed at one end) or Melting Point Tube.

**The 502-156 Fluorhib must be baked-off at 1000 °C for 10 minutes. It can then be stored up to 24 hours in a desiccators prior to use without being re-baked. The 781-335 Quartz Boats and 782-059 Nickel Liners or 625-505-430 Nickel Boat should be baked-off at 1000 °C for 5 minutes and stored in a desiccator until used.*

Calibration Samples

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

Method Parameters

Analysis Parameters

Carrier Gas	Oxygen
Purge Flow	3.00 lpm
Analysis Flow	0.75 lpm
Catalyst Heater	850 °C
Afterburner	850 °C

Element Parameters	*Carbon*	*Water*
Analyze	No	Yes
Conversion Factor	1.00	1.00
Significant Digits	5	5

Carbon Range	Auto
Switch Level to High Cell	34000
Switch back Level to Low Cell	28000
IR Baseline Time (seconds)	2
Endline Time (seconds)	2

Calibration Parameters

Water

Furnace Steps–Calcium Oxalate

Name	*Target*	*Ramp*	*Hold*	*Estimated Time*
Water	200	N/A	Calcium Oxalate	250-1000

Hold Parameters–Calcium Oxalate

	Water
Minimum Analysis Time (s)	250
Peak Threshold	0
Comparator Level	0.30
Maximum Analysis Time (s)	1000

Furnace Steps–Welding Flux

Name	*Target*	*Ramp*	*Hold*	*Estimated Time*
Free Moisture	105	N/A	360	360
Crystalline	1000	120.00	300	1060

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Determine blank–Standard.
   a. Enter 1.0000 g mass into Sample Login (F3) using blank as the sample name.
   b. Place a previously baked 782-059 Nickel Liner into a previously baked 781-335 Quartz Boat or use a 625-505-430 Nickel Boat.
   c. Place the boat on the furnace shelf (or appropriate autoloader position if so equipped) and initiate analysis (F5).
   d. Repeat steps 2a through 2c three to five times.
   e. Enter blank following procedure outlined in operator's instruction manual.
3. Determine blank–Sample.
   a. Enter 1.0000 g mass into Sample Login (F3) using blank as the sample name.
   b. Place a previously baked 782-059 Nickel Liner into a previously baked 781-335 Quartz Boat or use a 625-505-430 Nickel Boat.
   c. Add ~ 3 g 502-156 Fluorhib layered in nickel liner or nickel boat.
   d. Place the boat on the furnace shelf (or appropriate autoloader position if so equipped), and initiate analysis (F5).
   e. Repeat steps 2a through 2d three to five times.
   f. Enter blank following procedure outlined in operator's instruction manual.
4. Calibrate/Drift–Furnace Steps–Calcium Oxalate.
   a. Place a previously baked 782-059 Nickel Liner into a previously baked 781-335 Quartz Boat or use a 625-505-430 Nickel Boat.
   b. Weigh ~0.075 g of the reference material into boat and enter mass and sample identification into Sample Login (F3) for moisture calibration.

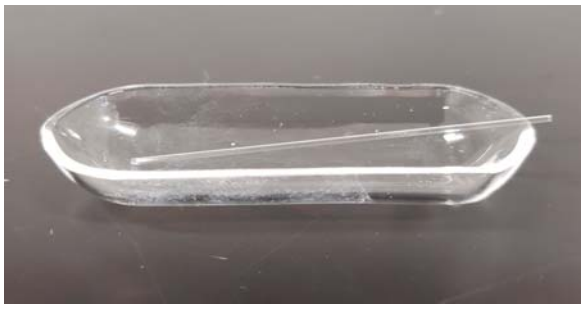
c. Place the boat on the furnace shelf (or appropriate autoloader position if so equipped), and initiate analysis (F5).
d. Repeat steps 3a through 3c three to five times for each calibration sample intended for calibration/drift.
e. Calibrate/drift using the procedure outlined in the operator's instruction manual.

5. Calibration Verification–Furnace Steps–Calcium Oxalate.
   a. Place a conditioned capillary/melting point tube into a previously baked 781-335 Quartz Crucible or 625-505-430 Nickel Boat. The capillary/melting point tube should be sealed on one end.
   b. Weigh ~5 mg (5 µL) of distilled or deionized water into the capillary/melting point tube.
   c. Enter mass, to the nearest 0.1 mg, and sample identification into Sample Login (F3).
   d. Without delay, place the boat on the furnace shelf (or appropriate autoloader position if so equipped), and initiate analysis (F5). Ensure that the open end of the capillary/melting point tube is resting above the top of the crucible and facing toward the boat stop, prior to initiating the analysis sequence. Please see the photos below for additional details.

Note: Per AWS A4.4M, a percent recovery of 95-105% is required for the system to be considered verified and analysis of samples may proceed.

6. Analyze Samples–Furnace Steps–Welding Flux
   a. Place a previously baked 782-059 Nickel Liner into a previously baked 781-335 Quartz Boat or use a 625-505-430 Nickel Boat.
   b. Add ~1.5 g 502-156 Fluorhib layered in bottom of nickel liner or nickel boat.
   c. Weigh ~4.000 g sample into boat and enter mass and sample identification into Sample Login (F3).
   d. Add ~1.5 g 502-156 Fluorhib covering the sample. Place the boat on the furnace shelf (or appropriate autoloader position if so equipped), and initiate analysis (F5).

Step 5d with Nickel Boat



Step 5d with Quartz Boat

*Typical Results**

Sample	*Mass (g)*	*% Free Moisture*	*% Crystalline H_2O*	*% Total H_2O*
Welding Flux	3.9994	0.041	0.092	0.133
	4.0007	0.040	0.087	0.127
	3.9971	0.041	0.092	0.133
	4.0002	0.041	0.089	0.130
	3.9972	0.042	0.092	0.134
	Avg =	***0.041***	***0.090***	***0.131***
	s =	***0.001***	***0.002***	***0.003***

**Calibrated with LECO 502-926 Calcium Oxalate Reference Material using linear forced through origin calibration.*

LECO Corporation | 3000 Lakeview Avenue | St. Joseph, MI 49085 | Phone: 800-292-6141 | 269-985-5496
info@leco.com • www.leco.com | ISO-9001:2015 Q-994 | *LECO is a registered trademark of LECO Corporation.*
LCRM = LECO Certified Reference Material; LRM = LECO Reference Material and are registered trademarks of LECO Corporation.

