Application Note

Instrument: 812 Series

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Determination of Moisture in Welding Flux

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Introduction

Welding flux provides several important functions during the welding process. The flux can assist with arc initiation and stability, particularly in AC welding applications. Under welding temperatures, the flux decomposes and generates shielding gases that protect the molten weld pool from atmospheric contamination, reducing oxidation and other negative reactions. Flux constituents also melt to form a slag layer that floats on the weld pool surface, protecting the molten weld metal from exposure to the atmosphere. The slag can further act as a thermal insulator, slowing weld cooling. For many steel welds, excessively rapid cooling may promote the formation of hard, brittle microstructures and increase cracking susceptibility.

Moisture absorbed by the flux can be a source of diffusible hydrogen in the weld metal and may contribute to hydrogen embrittlement. As a result, flux moisture content is commonly monitored as an important indicator of consumable performance and potential weld quality.

Instrument Model and Configuration

The LECO 812 Series is a line of multiphase carbon and moisture determinators with a variable temperature, ramping furnace and a high sensitivity detection system that is specifically designed to determine moisture in weld flux and other materials. The amount of moisture present in the weld flux is quantified by infrared measurement of H_2O formed from gases evolved during sample heating. The LECO M812 is a multiphase moisture-determination-only variant, and the CM812 adds simultaneous carbon determination, expanding system capability.

Method Reference

AWS A4.4M: Standard Procedures for Determination of Moisture Content of Welding Fluxes and Welding Electrode Flux Coverings

Accessories

781-335 Quartz Combustion Boats with 782-059 Nickel Boat Liners, or 625-505-430 Nickel Combustion Boats; 502-156 Fluorhib (baked-off)*; 501-171-HAZ Anhydrone; Capillary Tube or Melting Point Tube

**In accordance with AWS A4.4M, the 502-156 Fluorhib must be baked off at 1000 °C for 10 minutes and stored in a desiccator containing 501-171-HAZ anhydrone until use. If the baked Fluorhib is not used within twenty-four hours, it should be re-baked.*

Note: The 781-335 Quartz Combustion Boats and the 782-059 Nickel Boat Liners, or the 625-505-430 Nickel Combustion Boats should be pre-baked at 1100 °C for five minutes to remove any residual carbon then cooled in a desiccator until time of analysis. If the combustion boats are not used within twenty-four hours, they should be re-baked. After baking, handle combustion boats with clean tongs only; do not use fingers.

Reference Materials

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

Note: These reference materials require different furnace parameters than those used for moisture determination (refer to Furnace Step Method Parameters listed below).

Sample Preparation

A representative, uniform sample is required. Samples should be prepared in accordance with AWS A4.4M. Reference materials should be prepared according to the sample preparation statement on the certificate. Please reference the appropriate Safety Data Sheets (SDS) for safe handling of all reference materials and samples.

Method Parameters

Method Parameters: Moisture Determination

General Parameters	
Advanced Gas Flow	No
Purge Flow	4.00 LPM
Purge Time	15 s
Analytical Flow	0.75 LPM
Check Delay	20 s
Nominal Mass	1.0000 g
Manual Loading Mode	Dual Button
Afterburner Temperature	850 °C
Reagent Heater Temperature	High

Element Parameters	*Moisture*
Wait for Baseline Stability	Yes
Integration Delay	0 s
Starting Baseline	2 s
Integrate from Furnace Method	Yes
Use Endline	Yes
Ending Baseline	2 s

Furnace Method Parameter

Furnace Method Parameters: Calibration

Apply Peak Find to This Furnace Method	No
Name	Calibration
Gas	Oxygen
Peak Monitor	On
Analyte	Moisture
Moisture Threshold Level	0.30
Minimum Time	120 s
Maximum Time	600 s
Mode	Constant
Temperature	200 °C

Furnace Method Parameters: Weld Flux Analysis

Apply Peak Find to This Furnace Method	Yes

Step 1: Free Moisture Determination

Name	Free Moisture
Gas	Oxygen
Peak Monitor	Off
Mode	Constant
Temperature	105 °C
Hold Time	360 s

Step 2: Crystalline Moisture Determination Ramping Parameters

Name	Crystalline Moisture
Gas	Oxygen
Peak Monitor	Off
Mode	Ramped
Target Temperature	1000 °C
Ramp Rate	120 °C/min

Step 3: Crystalline Moisture Determination Hold Time Parameters

Name	Hold
Gas	Oxygen
Peak Monitor	Off
Mode	Constant
Hold Time	300 s

Peak Find Settings

Strategy	User Specified
Peaks	2

Peak 1 – Free Moisture

Name	Free Moisture
Peak Start	Fixed Time
Start Time	0.1 seconds
Peak End	Fixed Length
Peak Length	360.0 seconds

Peak 2 – Crystalline Moisture

Name	Crystalline Moisture
Peak Start	End of Previous Peak
Peak End	End of Sample

Procedure

1. Prepare the instrument as outlined in the operator's instruction manual.
2. Determine calibration blank.
   a. From the Analysis Screen, use the Login Bar to add three or more blank replicates.
   b. Select "Moisture Determination" as the Method, and "Calibration" as the Furnace Step Method (parameters noted above).
   c. Place a pre-baked combustion boat on the shelf directly in front of the closed combustion furnace door (for manual loading systems) or into the appropriate position in the autoloader.
   d. For manual loading systems, initiate the analysis sequence. When prompted by the software, press the Analyze button and load the combustion boat into the combustion tube until it reaches the boat stop.
   e. For manual loading systems, when analysis is complete, remove the combustion boat and close the combustion furnace door.
   f. For manual loading systems, perform steps 2a through 2e a minimum of three times.
   g. For auto-loading systems, perform steps 2a through 2c a minimum of three times and initiate the analysis sequence.
   h. Set the blank for the reference materials following the procedure outlined in the operator's instruction manual.
3. Calibration.
   a. Weigh a suitable mass of an appropriate reference material into a pre-baked combustion boat. Enter the reference material mass and identification into the Analysis Screen.
   b. Select "Moisture Determination" as the Method and "Calibration" as the Furnace Method (parameters noted above).
   c. Place the combustion boat containing the reference material on the shelf directly in front of the closed combustion furnace door or into the appropriate position in the autoloader.
   d. For manual loading systems, initiate the analysis sequence. When prompted by the software, press the Analyze button and load the combustion boat into the combustion tube until it reaches the boat stop.
   e. For manual loading systems, when the analysis is complete, remove the combustion boat and close the combustion furnace door.
   f. For manual loading systems, perform steps 3a through 3e a minimum of three times for each reference material used.
   g. For auto-loading systems, perform steps 3a through 3c a minimum of three times for each reference material used and initiate the analysis sequence.
   h. Calibrate the instrument following the procedure outlined in the operator's instruction manual.
4. Calibration Verification.
   a. Place a conditioned capillary/melting point tube into a pre-baked combustion boat.
   b. Weigh ~5.0 mg (5 µL) of distilled or deionized water into the capillary/melting point tube.
   c. Enter the mass (to the nearest 0.1 mg) and the sample identification into the Analysis Screen.
   d. Select "Moisture Determination" as the Method and "Calibration" as the Furnace Step Method (parameters noted above).

e. Without delay, place the combustion boat containing the capillary/melting point tube on the shelf directly in front of the closed combustion furnace door or into the appropriate position in the autoloader.
f. For manual loading systems, initiate the analysis sequence. When prompted by the software, press the Analyze button and load the combustion boat into the combustion tube until it reaches the boat stop.
g. For manual loading systems, when the analysis is complete, remove the combustion boat and close the combustion furnace door.
h. For auto-loading systems, initiate the analysis sequence.

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

5. Determine sample blank.
   a. From the Analysis Screen, use the Login Bar to add three or more blank replicates.
   b. Select "Moisture Determination" as the Method and "Weld Flux Analysis" as the Furnace Step Method (parameters noted above).
   c. Weigh ~3.0 g of baked-off 502-156 Fluorhib into a pre-baked combustion boat.
   d. Analyze the sample blank replicates following steps 2c through 2g a minimum of three times.
   e. Set the blank for the samples following the procedure outlined in the operator's instruction manual.
6. Analyze samples.
   a. Weigh ~1.5 g of baked-off 502-156 Fluorhib into the bottom of a pre-baked combustion boat and spread the material into an even layer within the bottom of the combustion boat.
   b. Weigh an appropriate mass of sample (refer to the AWS A4.4M method and the table below for recommended sample masses) into the combustion boat on top of the Fluorhib. Enter the sample mass and identification into the Analysis Screen.

Recommended Sample Mass for Analysis per AWS A4.4M	
Expected Water (%)	*Nominal Sample Mass (g)*
<0.20	5.0
0.21 - 1.00	2.0
0.51 – 1.00	1.0
1.00	0.5

   c. Weigh ~1.5 g of baked-off 502-156 Fluorhib into the combustion boat covering the sample.
   d. Select "Moisture Determination" as the Method and "Weld Flux Analysis" as the Furnace Step Method (parameters noted above).
   e. Place the combustion boat containing the sample and Fluorhib on the shelf directly in front of the closed combustion furnace door or into the appropriate position in the autoloader.
   f. For manual loading systems, initiate the analysis sequence. When prompted by the software, press the Analyze button and load the combustion boat into the combustion tube until it reaches the boat stop.
   g. For manual loading systems, when analysis is complete, remove the combustion boat and close the combustion furnace door.
   h. For manual loading systems, perform steps 6a through 6g for each sample to be analyzed.
   i. For auto-loading systems, perform steps 6a through 6e for each sample to be analyzed and initiate the analysis sequence.

Typical Data

The moisture calibration was performed utilizing a linear, force through origin calibration using ~0.075 g of LECO 502-926 (Lot 1007) Calcium Oxalate LCRM (12.1 % Moisture). The calibration was verified by analyzing a capillary/melting point tube containing ~5.0 mg (5 µL) of distilled water and confirming that a moisture recovery of 95-105% moisture was achieved (as per AWS A4.4M). The Calcium Oxalate was dried at 105 °C for one hour prior to analysis and stored in a desiccator until use (within 24 hours).

Sample	*Mass (g)*	*Moisture (%)*	*Free Moisture (%)*	*Crystalline Moisture (%)*
Welding Flux 880 Beige	4.0488	0.179	0.045	0.134
	4.0067	0.177	0.056	0.121
	4.0038	0.178	0.049	0.129
	4.0307	0.179	0.052	0.127
	3.9901	0.174	0.050	0.124
	$\bar{x}$ =	***0.178***	***0.051***	***0.127***
	s =	***0.002***	***0.004***	***0.005***
Welding Flux 780 Brown	4.0025	0.061	0.015	0.046
	4.0074	0.062	0.017	0.045
	4.0049	0.065	0.016	0.049
	4.0034	0.067	0.016	0.050
	4.0035	0.063	0.015	0.048
	$\bar{x}$ =	***0.064***	***0.016***	***0.048***
	s =	***0.002***	***0.001***	***0.002***
E7018 Weld Flux	1.0149	0.545	0.140	0.405
	1.0095	0.536	0.143	0.392
	1.0044	0.540	0.143	0.397
	1.0013	0.529	0.134	0.395
	1.0482	0.531	0.137	0.394
	$\bar{x}$ =	***0.536***	***0.139***	***0.397***
	s =	***0.007***	***0.004***	***0.005***

$\bar{x}$ = Sample Mean; s = Sample Standard Deviation

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