



OPERATING MANUAL

HOT FOIL™ LOI INSTRUMENT

A LABORATORY INSTRUMENT TO DETERMINE
THE LOI VALUE OF FLY ASH

DESIGNED AND MANUFACTURED* BY:

Fossil Energy Research Corp.
Laguna Hills, California

*Manufacturing License Granted by the Electric Power Research Institute

10/08

HOT FOIL™ LOI INSTRUMENT

TESTING SPECS AT A GLANCE

Dry:

To drive off any moisture in sample prior to weighing.

Test:

Heats sample to approximately 750°C per ASTM specification.

Foil Time:

Same for dry and test. Internally set by factory, not user adjustable.

INSTRUMENT SPECIFICATIONS

Sample Parameters:

Ash sample size: 20-80 mg

Test time: Three repeat LOI determinations, 30 minutes

Accuracy/Reproducibility

To be demonstrated.

Utilities/Supplies

Electrical: 120 VAC, maximum 2 amps continuous, 5 amps transient

Environment

Temperature: 50-90°F

Humidity: Avoid Extremes

Cabinet

Dimensions: 15W x 15D x 8 H Inches

Weight: 35 lbs (16 Kg)

PARTS LIST

Enclosed in Initial Shipment

HOT FOIL™ LOI Instrument Assembly (1)
Tweezers (1)
Test Foils (1 set of 5)
Foil Stands, Acrylic (2)
Brush (2)
Scupula (1)
Calculation Pad (1)
Breeze Shield (1)
HOT FOIL LOI Instrument Instruction Manual (1)
Spare Fuse, 5 amp slow blow (MDL-5)

Also Available from FERCo

Semi-Microbalance (± 0.01 mg readability; ± 0.02 mg repeatability) (1)

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1 INSTALLATION

1.1 Visual Inspection

The HOT FOIL™ LOI Instrument and accompanying accessories should be visually inspected as soon as they are unpacked to ensure that all items have been received and none has sustained physical damage. The carrier and your FERCo representative should be notified immediately if shipping damage is found. An inspection report must be obtained from the carrier for insurance purposes if damage due to shipment has occurred.

1.2 Location

The instrument should be installed on a flat surface with adequate space on all sides to permit easy access to the test clips. The balance should be placed on a stable surface suitable for precision weighing. The instrument and balance should be located 1) in close proximity to each other for ease in transferring samples between weighings, and 2) in a draft-free area to prevent loss of any ash sample during transfer and to ensure accurate weighings.

1.3 Electrical Requirement

Instruments are shipped to operate at 120 VAC. Maximum current draw is 2 amps with transient peaks of 5 amps.

2 GENERAL DESCRIPTION

2.1 Principle of Operation

The HOT FOIL™ LOI Instrument was built by Fossil Energy Research Corp. (FERCo). The technique uses a small quantity of ash (20 to 80 mg) applied to a metal foil that is electrically heated. Upon heating, compounds are driven from the ash simulating the ASTM LOI measurement. The sample weight loss is divided by the initial weight of the sample to determine the LOI. Although the methodology does not physically simulate the ASTM LOI procedure, the HOT FOIL™ LOI Instrument measurements have been demonstrated to yield the same LOI values as those determined for the same ash samples utilizing the ASTM procedure.

3 OPERATION

3.1 Start Up/Set Up

Refer to Section 1 for proper installation of the HOT FOIL™ LOI Instrument and analytical balance. The analytical balance must be properly warmed up before accurate measurements can be made (refer to the Owner's Manual for manufacturer specifications). The power switch for the HOT FOIL™ LOI Instrument is located on the back panel. The instrument requires no warm up. However, prior to the first test, the foil setting should be confirmed. Meter setting for the “dry” cycle should be “3”, meter setting for the “test” cycle should be “10”. These are adjusted by the corresponding rheostats on the front panel with the foil energized.

When the HOT FOIL™ LOI Instrument's power switch is turned on, the meter will display numbers and either the “test” or “dry” indicator will be lit, and the fan will turn on. These are the only assured indications that the power is on. Turning the power switch off will abort a test-in-progress and reset the internal timer.

A breeze guard is provided to cover the foil during the test. This guard also protects the user from accidental contact with the foil. The breeze guard must be in position before the HOT FOIL™ LOI Instrument will operate and must remain in position for the entire heating cycle.

A hot energized foil is indicated by the red energize light and an audio alarm.

3.2 LOI/FOIL Test Procedure

Start here if beginning with a brand new test foil, otherwise, skip to Step 3

- 1) Testing foils, pre-folded into a boat configuration are available from FERCo (Part Number HF 440).
- 2) To prepare the foil to hold sample, heat the foil in the “test” mode with the meter set higher than normal (i.e., “11”). This “pre-burn” should burn off any oils or particles left on

the foil from handling. **From this point until the end of the test, use only the tweezers to handle the foil.**

- 3) In general, when installing the foil in the instrument, first install the foil in stationary clip, then bring movable clip into position and secure. Use the tweezers to hold/steady/guide the foil into position since it does have some spring. This is critical when an ash sample is being positioned.

From this point, use only tweezers to handle the foil!

- 4) When the pre-burn is done, allow a few moments for the foil to cool. **Using the tweezers**, lift the foil from the clamps and position in the support block within the balance. Measure and log the foil weight. Be sure to “tare out” weight of foil support block. (Refer to balance manual for proper operation and calibration.)
- 5) Place the foil in a second support block. Shake and mix the LOI sample bag well to ensure a representative sample. Scoop 20 to 80 mg of sample into the “V” of the foil (with practice, you will learn to “eyeball” this very well). Use of a small brush helps in both loading and distributing the sample within the foil. Use the brush to remove any extra sample on the outside of the foil. Press “Tare” on the scale (do this before **every** weight measurement) and measure and log the foil + sample weight.
- 6) NOTE: Moving the foil without losing sample is tricky - this will take practice. Losing sample will necessitate a re-weighing of the sample at best, or starting the analysis over at worst.

If drying the sample is unnecessary, skip to Step 9.

- 7) **Carefully** reattach the foil to the clips and replace breeze guard. Press the “dry” button to engage the drying mode. Depress “energize” button to pass current through foil; the “energize” button will remain lit while current flows through the foil. Adjust “dry” meter setting to “3”. System will turn off after the predetermined cycle time.

- 8) Detach foil and dried sample. Carefully transfer to balance. Measure and log the foil + dry sample weight.
- 9) **Carefully** reattach the foil to the clips and replace breeze guard. Press the “test” button to engage the test mode. Depress the “energize” button to pass current through the foil; the “energize” button will remain lit while current flows through the foil. Adjust “test” meter setting to “10”. System will turn off after the predetermined cycle time.

NOTE: If LOI value calculated below is greater than 12%, retest the sample at a slightly higher meter setting (“10.3” to “10.5”) to ensure complete burn off. Return test setting to “10” with LOI values less than 12%.

If LOI value exceeds 20%, subject the sample to two test cycles before weighing (if samples are consistently greater than 20% LOI, contact Fossil Energy Research for information on resetting the internal timer).

- 10) Allow a few moments for foil and clips to cool. Detach foil and sample. Carefully transfer to balance; measure and log the foil +Ash Residue sample weight.
- 11) The ash residue sample may be thrown away. Clean the foil using a tissue, brush, or a blast of air. Inspect the foil for holes or cracks where sample could fall out. Reshape the foil as necessary using the “scoop” or your fingers (refer to Section 4). After several uses, a foil may begin cracking at the fold at the ends. This is OK, as long as the foil will hold sample. Normally, a foil will hold up for 20-30 tests. Go back to Step 4 to perform another test. If foil was touched, return to Step 2.
- 12) If foil “sticks” to clips, use a fine grit emery cloth or equivalent to clean the clip surface. Be sure meter setting is not too high (“10” to “10.5”). Refer to Section 4 for more discussion.

3.3 LOI Calculation

To get the LOI % number, calculate the following:

$$\text{Foil Weight} = [A]$$

$$\text{Foil} + \text{Wet Sample Weight} = [B]$$

$$\text{Foil} + \text{Dry Sample Weight} = [C]$$

$$\text{Foil} + \text{Ash Residue Sample Weight} = [D]$$

$$\text{Dry Sample Weight} = [E] = [C] - [A]$$

$$\text{Ash Residue Sample Weight} = [F] = [D] - [A]$$

$$\text{LOI \%} = \frac{[E] - [F]}{[E]} \times 100$$

3.4 Shut Down

Shut down the balance according to the manufacturer's specification. The HOT FOIL™ LOI Instrument requires no special shut down procedure other than turning off the main power switch.

4 OPERATIONAL TIPS

1. Oxidizing a virgin foil for the first time results in a small shiny region at each end of the foil where the clips are attached. The life of the foil can be extended and foil sticking minimized if the foil is consistently gripped on this shiny surface.
2. After extended use, a particular foil may begin sticking to one or the other clip after the high current test cycle. Freeing a stuck clip generally results in spilled sample and an aborted test. The sticking is a result of localized spot welding of foil to the clip due to a localized high current density. Once a foil sticks, a small deposit is usually left on the clip which exacerbates the localized high current density and results in a stuck foil in subsequent tests.

After a foil sticks, the gripping surface of the clip should be cleaned to remove any surface imperfections. This is best accomplished with a folded 400 grit emery paper or silicon carbide abrasive paper (or equivalent) positioned in the clip and slid against the gripping surfaces while the clip's spring tension holds the gripping surface against the abrasive. A few strokes will remove imperfections and result in a clean gripping surface.

3. While the gripping surface of the clip can be cleaned, little can be done to dress the foil ends. Every sticking occurrence results in a loss of foil material and a surface discontinuity which will promote further sticking incidents. At the extreme, the gripped ends of the foils will have a series of visible pin holes. Dressing the clip will minimize the sticking; however, a foil that has ragged ends will continually pose a sticking problem. Do not hesitate to dispose of old foils.
4. During continued use, the "V" opening of the foil boat will likely begin to close. In general, this should not be a problem as long as sample can still be placed in the "V". If the gap becomes too narrow, use the scupula to open the "V". Do not open too wide; included angle should be no more than 60°. Repeated opening of the "V" will result in

fatigue cracks at the bottom fold and subsequent loss of sample. Be very careful when using your fingers (foil is sharp) and be sure to heat the foil afterward to burn off any oils from your fingers.

5. During normal operation, the insulating pads on the clips should be sufficient to permit immediate grasping and release of the foil. A clip that is hot to the touch immediately after a test is not normal and is an indication of excessive resistance heating between the clip and foil. The contact resistance is a result of an oxidized foil (refer to Paragraph 1), oxidized gripping surface, or both. An overly oxidized foil can lead to foil sticking and should be replaced. The gripping surface can be cleaned as described in Paragraph 3.
6. The spring loaded movable clip is designed to offset the thermal growth of the foil during a test, keeping the foil relatively taut. The tensile load on the foil should be no more than necessary to keep the foil from buckling during a test. Excessive tensile load causes the foil's "V" to close and can result in the clip slipping off foil during a test.
7. The movable clip should slide freely and smoothly. Periodically, a light coating of lubricant (silicone, light oil, etc.) on the clip is recommended to insure smooth operation.
8. When installing the foil, first place the foil in the stationary clip and then bring the movable clip into position guiding the foil to the gripping surface. Spring tension should pull the foil taut when your hand is released. If necessary, reposition the foil in the stationary clip by releasing its grip. Do not apply excessive force to the foil. This may result in foil buckling or impart a "spring" the foil which may result in sample loss when the clips are released.

When removing the foil, slide the movable clip slightly to release the tension on the foil before opening the jaws. Slide clip back to clear the foil. Use tweezers as necessary to guide the foil into its relaxed position. Release the jaws on the stationary clip.

9. The foils have a spring-like nature. They can take a set and if released without guidance, can result in lost sample. Take care when handling the foil.

10. Successful handling of the foil and valid tests may require some practice. The spring-like nature of the foil can be frustrating but repeated usage and familiarity will result in meaningful results without problems.

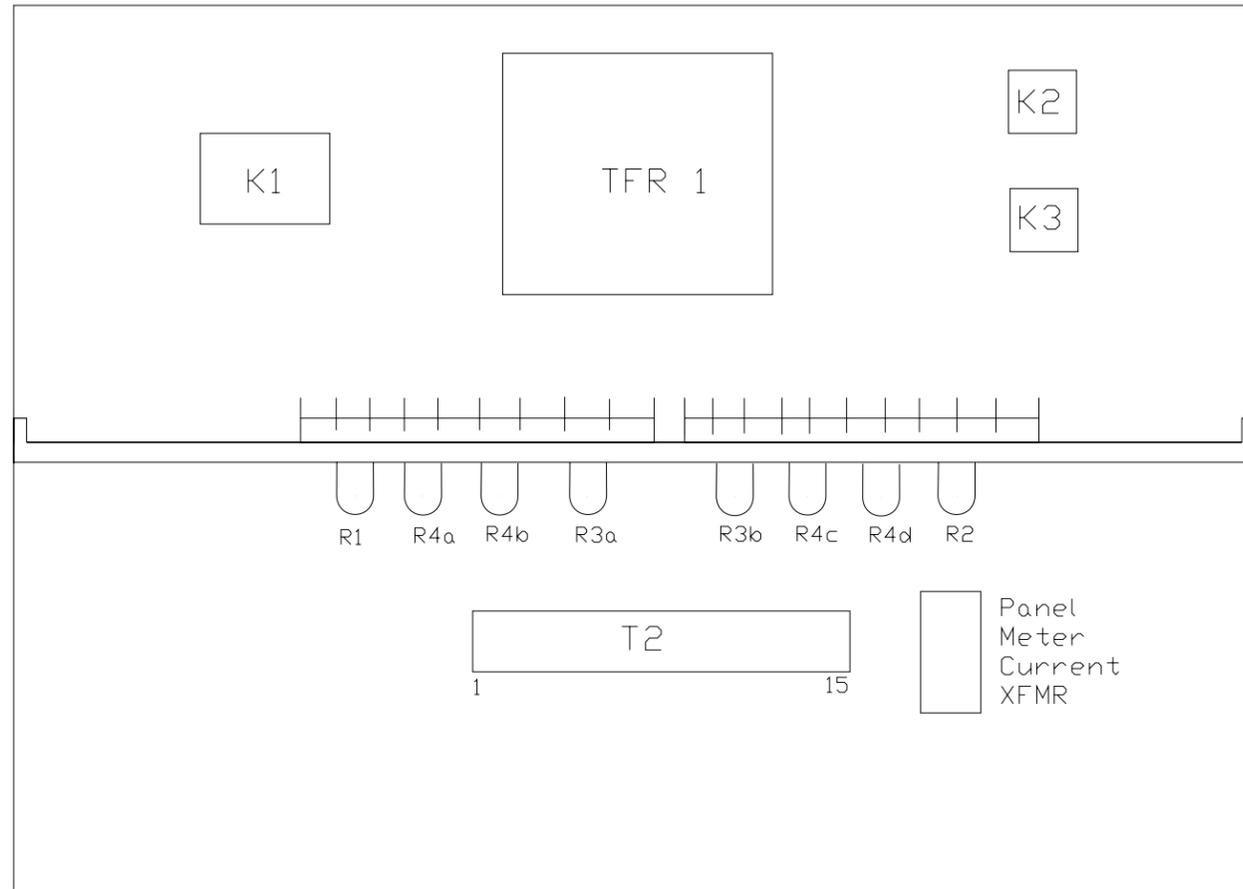
11. Should the fan filter become dirty or plugged over time, it can be cleaned by washing in a stream of water and drying with the compressed air. The filter is removed by gently prying apart the filter cover from under one of the side tabs with a small flat-head screwdriver. Upon replacing the cleaned filter, the cover is replaced by snapping it into place under the tabs on the sides of the filter housing.

5 TROUBLESHOOTING

The HOT FOIL™ LOI instrument is, in general, quite rugged and can survive normal handling and shipping while providing trouble-free services. However, if the HOT FOIL™ LOI instrument does not operate properly, please refer to the following table for possible solutions. If the instrument still fails to operate properly after attempting the “fixes” listed below, please contact Fossil Energy Research Corp.

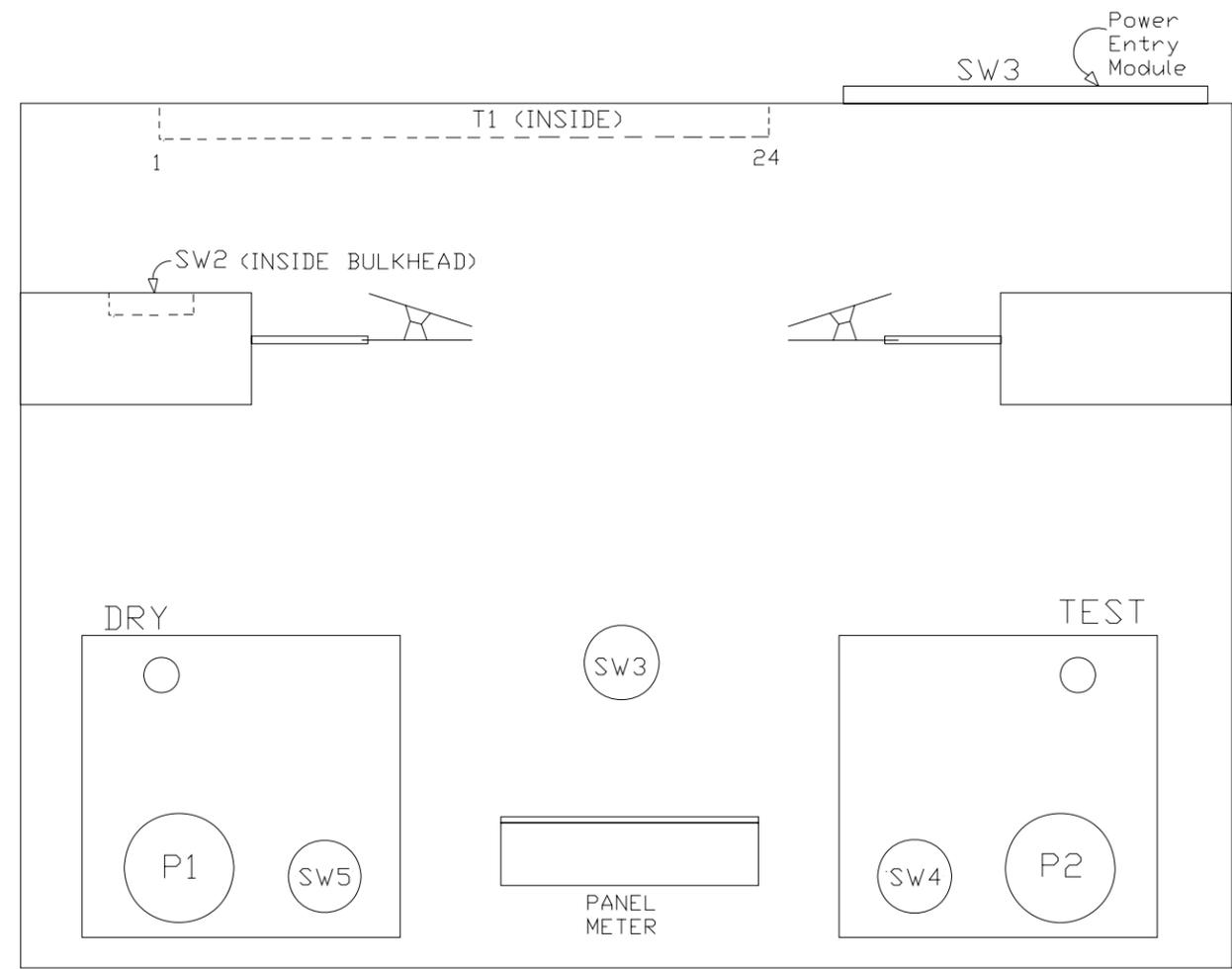
Problem	Check For:
<p>1. Instrument will not power up (i.e., no digital display, one amber light not illuminated).</p>	<p>A. Instrument cord plugged into a “live” 115 VAC circuit.</p> <p>B. Proper fuse installation in power entry module. (Proper installation has fuse to TOP of instrument.)</p> <p>C. Integrity of fuse (is it blown?).</p> <p>D. Internal wiring disconnection. (UNPLUG INSTRUMENT. Remove 12 Phillips head screws around instrument to separate the white upper shell from the gray lower shell. Upper shell pivots to the back of instrument.)</p>
<p>2. Amber light will not switch from “test” to “dry” (or vice versa) when push button is activated.</p>	<p>A. K2 or K3 relay being separated from its socket. (Open up instrument as described in 1-D.)</p>
<p>3. Digital display will not change from 0.0 when adjustment knobs are rotated.</p>	<p>A. The energized light being illuminated, indicating a test is in progress. The adjustment controls are only active when current is flowing through the foil as indicated by the illuminated energized light.</p> <p>B. The proper installation of a foil in the clips.</p> <p>C. Broken or disconnected power wires to the clips (accessible through the cap plugs on the foil clip bulk heads and/or by separating the upper and lower shells).</p>
<p>4. Energize light will not illuminate when button is pushed.</p>	<p>A. Proper installation of the breeze shield. The black magnetic actuator MUST BE ON THE LEFT SIDE OF THE INSTRUMENT.</p>

Problem	Check For:
	B. Proper settings on the K1 relay. Separate upper and lower shells as described in 1-D. The thumbwheel switches should read "C120S".
5. Digital display does not read "0.0" when foil is not energized.	<p>A. A small fraction of displays have exhibited the tendency for the zero to drift as they age. Treat the non-zero reading as an offset and adjust current accordingly (i.e., if display shows 0.5 in the de-energized mode, adjust to 3.5 or 10.5 for dry or test modes). The offset does not appear to affect the accuracy of the display if the aforementioned considerations are taken.</p> <p>If the display exhibits this behavior within the one-year warranty period, return to Fossil Energy Research for replacement.</p>



FRONT

BOTTOM PANEL
Component Layout



FRONT

TOP PANEL
Component Layout