

# Gross Heat in Fuel Oils and Biomass

LECO Corporation; Saint Joseph, Michigan USA

## Instrument: AC600

### Introduction

The gross calorific value of Biomass materials is most often used when calculating the total calorific value for a quantity of Biomass materials being used for fuel purposes. Gross calorific value of fuel oils and other liquid hydrocarbon fuels are used in calculating the thermal efficiency of equipment and calculating the mass and or volumetric heat of combustion for the fuel.

### Sample Preparation

A representative, uniform sample is required.

### Accessories

774-204 Crucible, Syringe & Needle, pressure sensitive tape (cellophane tape 38 mm or 1.5 inch wide that is sulfur and chlorine free), disposable eyedroppers, LECO 502-815 Mineral Oil for spiking Biomass sample, 776-978 Glass Scoop.

### Calibration Samples

Benzoic acid pellets made from NIST 39i, or LECO 774-208 Benzoic Acid Pellets.

### Method Parameters

Method	TruSpeed™
Standard Mode	ASTM D5865-13

### Thermochemical Corrections

Titrant Energy Value	0.0039683 Btu/ml
Sulfur Correction	23.861 Btu/lb
Calculation Mode	TruSpeed
Analysis Time	4.75 minutes
Equilibrate Time	1.5 minutes
Pre-Fire Time	0.5 minutes
Main Time	2.75 minutes
Stir Speed	13.0

### System Parameters - Database

Fuse Type	Cotton
Fuse Length	10 cm
Fuse Combustion Heat	0.006255562 Btu/cm
Significant Digits	5
Result Units	Btu/lb
Sleep Timeout	90 minutes
Water Temperature	25°C
Auto Increment Sample Name	Disable
Alarm	Yes



### Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Choose the TruSpeed method for analysis.
3. Condition the system by analyzing one LECO 774-208 Benzoic Acid Pellet.
4. Calibration
  - a) Calibrate the instrument using a minimum of five analyses of 1 gram Benzoic Acid Pellets, following the procedure outlined in the operator's instruction manual.
5. Analyze Samples: Residual, Mineral, and Bio-Oil
  - a) Place 774-204 Crucible on balance and tare.
  - b) Weigh ~0.6 g sample into crucible using a disposable eyedropper.
  - c) Enter mass and sample identification into Sample Login (F3).
  - d) Place crucible in "crucible holder" and tie cotton thread fuse to the igniter wire, making sure the cotton thread fuse contacts the oil sample.
  - e) Assemble the combustion vessel and pressurize following the procedure outlined in the operator's instruction manual.
  - f) Transfer combustion vessel to instrument and analyze.
  - g) Perform the appropriate corrections for sulfur and nitrogen.
6. Analyze Samples: Diesel Fuel, Kerosene, Jet Fuel, and Gasoline

*Note: This group of highly volatile fluids requires the use of cellophane tape to reduce sample loss during weighing and analysis. It is recommended to prevent damage to the vessel to analyze lower amounts of tape than the ASTM method due to the varying calorific values of cellophane tape. Refer to ASTM D240 for additional details.*

- a) Place 774-204 Crucible on balance and tare.
- b) Determine the calorific value of the pressure sensitive (Scotch) tape by analyzing 0.6 g of tape in crucible.
- c) Repeat step 6b three to five times and use the average as the "spike value".
- d) Enter spike value (Btu/lb) in Method Parameters.
- e) Place 774-204 Crucible on balance and tare.
- f) Remove crucible from balance and place a piece of pressure sensitive (Scotch) tape across the top of crucible. Trim around edge with razor blade or scissors and seal tightly. See photos on page 2.

- g) Place a approximate 3 x 12 mm strip of tape creased in the middle and sealed by one edge in the center of the tape disk to create a flap on top of the crucible.
- h) Place crucible on balance, record weight of tape and tare.
- i) Enter weight of tape as "spike weight".
- j) Using a syringe and needle, add ~0.6 g sample to the crucible by inserting the needle through the tape disk at a point so that the flap of tape will cover the puncture upon removal of needle. Seal down flap by pressing lightly.

*Note: Take care throughout weighing and filling operation to avoid contacting the tape or crucible with bare fingers.*

- k) Enter mass of sample and sample identification into Sample Login (F3).
- l) Place crucible in the "crucible holder"; tie cotton thread fuse to igniter wire, and arrange cotton thread fuse to contact/lie on top of the center of the tape disk.
- m) Assemble the combustion vessel and pressurize following the procedure outlined in the operator's instruction manual.
- n) Transfer the combustion vessel to the instrument and analyze.
- o) Perform appropriate corrections for sulfur and nitrogen.



**Step 6f**

## 7. Analyze samples: Biomass

**Note:** Biomass samples require the addition of a combustion aid (spiking agent) such as mineral oil. LECO 502-815 mineral oil has an assigned calorific value and can be used for this purpose. Other mineral oils may be used; however, the calorific value of the oil must be determined (refer to step 5 for analysis of mineral oil).

- a) Place 774-204 Crucible on balance and tare.
- b) Weigh ~0.2 to 0.4 g biomass sample into crucible.
- c) Enter mass and sample identification in sample login (F3)
- d) Tare crucible and add ~0.2 to 0.4 g 502-815 mineral oil as a spike using a disposable eyedropper.

*Note: Ratio of sample/spike is dependant on sample density (Total mass should be ~0.6 g).*

- e) Enter mass of mineral oil added in 7d as "Spike Weight".
- f) Let stand for 3 to 5 minutes or until oil is completely absorbed in sample.
- g) Enter spike value (Btu/IB of mineral oil) in Method Parameters.
- h) Place crucible in the "crucible holder", and tie cotton thread fuse to the igniter wire making sure the cotton thread fuse contacts the sample.
- i) Assemble combustion vessel and pressurize following the procedure outlined in the operator's instruction manual.
- j) Transfer the combustion vessel to the instrument and analyze.
- k) Perform appropriate corrections for sulfur, nitrogen, and moisture.



**Step 6f**



**Step 6g**



**Step 6j**

## Typical Results

Sample	Mass g	Btu/lb
Mineral Oil	0.6087	19800
	0.6052	19819
	0.6089	19809
	0.6098	19806
	0.6059	19812
	<b>X =</b>	<b>19809</b>
	<b>s =</b>	<b>7</b>
Bio Oil	0.6103	7445
	0.6056	7403
	0.6061	7369
	0.6047	7442
	0.6057	7419
	<b>X =</b>	<b>7416</b>
	<b>s =</b>	<b>31</b>
No. 2 Diesel Fuel	0.6201	19540
	0.6111	19531
	0.6078	19533
	0.6325	19514
	0.6282	19532
	<b>X =</b>	<b>19530</b>
	<b>s =</b>	<b>10</b>
No. 2 Diesel Fuel	0.6296	19409
	0.5763	19406
	0.6125	19406
	0.6168	19401
	0.6235	19423
	<b>X =</b>	<b>19409</b>
	<b>s =</b>	<b>8</b>
Wood Chip (Biomass) #1	0.2233	6697
	0.2292	6742
	0.2265	6753
	0.2267	6747
	<b>X =</b>	<b>6734</b>
	<b>s =</b>	<b>26</b>
Wood Chip (Biomass) #2	0.2221	8084
	0.2291	8110
	0.2283	8050
	0.2219	8107
	<b>X =</b>	<b>8087</b>
	<b>s =</b>	<b>28</b>