# Determination of Carbon, Hydrogen, and Nitrogen in Biomass

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## Instrument: CHN628

### *Introduction*

Carbon, Hydrogen, and Nitrogen determination is part of the ultimate analysis of solid fuel materials, helping to characterize the materials and provide information that can be utilized in calculating material/energy balances and efficiencies, as well as emissions potentials for the solid fuel materials.

The LECO CHN628 is a combustion elemental carbon, hydrogen, and nitrogen instrument that utilizes only pure oxygen in the furnace, ensuring complete combustion and superior recovery of the elements of interest. A combustion gas collection and handling system lowers the overall cost-per-analysis and extends reagent lifetimes. A carrier gas sweeps the combustion gas to separate infrared cells utilized for the detection of $H_2O$ and $CO_2$, while a thermal conductivity cell is used for the detection of nitrogen.

### *Sample Preparation*

A representative, uniform sample is required.

### *Accessories*

502-186 Tin Foil Cups, 501-441 Sucrose

### *Calibration Samples*

502-642 Phenylalanine, 502-092 EDTA, 501-050 Nicotinic Acid, or other suitable pure compounds.

### *Analysis Parameters*

| | |
|---|---|
| Combustion Temperature: | 950–1050°C |
| Afterburner Temperature: | 850°C |

### *Element Parameters*

| | *Carbon* | *Hydrogen* | *Nitrogen* |
|---|---|---|---|
| Analyze | Yes | Yes | Yes |
| Baseline Delay Time | 0 sec | 0 sec | 10 sec |
| Min. Analysis Time | 20 sec | 40 sec | 40 sec |
| Comparator Level | 100.00 | 100.00 | 100.00 |
| Endline Time | 1 sec | 1 sec | 2 sec |
| Conversion Factor | 1.00 | 1.00 | 1.00 |
| Significant Digits | 5 | 5 | 5 |
| IR Baseline Time | – | – | 1 sec |
| TC Baseline Time | – | – | 10 sec |

### *Burn Profile*

| *Burn Steps* | *Time (seconds)* | *Furnace Flow* |
|---|---|---|
| 1 | 40 seconds | High |
| 2 | 30 seconds | Medium |
| 3 | 30 seconds | High |

### *Ballast Parameters*

| | |
|---|---|
| Equilibrate Time | 30 seconds |
| Not Filled Timeout | 300 seconds |

### *Aliquot Loop*

| | |
|---|---|
| Fill Pressure Drop | 200 mm Hg |
| Equilibrate Pressure Time | 8 seconds |

### *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 (F3) using Blank as the sample name.
   b. Select 10 replicates.
   c. Initiate the analysis sequence (F5).
   d. Set the blank using the last five results following the procedure outlined in the operator's instruction manual.

   *Note: The standard deviation of the last five blanks should be less than or equal to 0.001% (10 ppm) for carbon and nitrogen, and less than or equal to 0.002% (20 ppm) for hydrogen. Additional blanks beyond the recommended 10 may be required in order to achieve the recommended precision.*
3. Calibrate
   a. Weigh 0.10 to 0.20 g of pure compound calibration sample (Phenylalanine, EDTA, Nicotinic Acid, etc.) into a 502-186 Tin Foil Cup and seal.
   b. Enter sample mass and identification into Sample Login (F3).
   c. Transfer tin foil cup containing the sample to the appropriate position of the sample carousel.
   d. Initiate the analysis sequence (F5).
   e. Repeat steps 3a through 3d a minimum of five times.
   f. Calibrate the instrument using single standard calibration (fixed at origin) following the procedure outlined in the operator's instruction manual.
   g. Verify the calibration by analyzing ~0.15 g of a different pure compound following steps 3a through 3d.

   *Note: Multi-point (fractional weight or multiple calibration samples) may be used to calibrate if desired. Typically single-point calibration using a pure compound provides a suitable and cost-effective calibration. Refer to the operator's instruction manual for details regarding multi-point calibration.*

Organic Application Note

Delivering the Right Results

4. Analyze Samples
   a. Weigh ~0.15 g of sample into a 502-186 Tin Foil Cup and seal.
   b. Enter mass and identification information into Sample Login (F3).
   c. Transfer tin foil cup containing the sample to the appropriate position of the sample carousel.
   d. Initiate the analysis sequence (F5).

   *Note: Carbon, Hydrogen, and Nitrogen results for Biomass samples are typically reported on a dry basis. Therefore, either the materials must be dried prior to analysis or the moisture content determined using a thermogravimetric analyzer and entered during the Sample Login procedure. Samples are typically dried at 85°C for two hours prior to analysis.*
5. Determining Atmospheric Blank (Some atmosphere will be trapped with the sample when it is encapsulated in the tin foil cup. This will cause biased high results at low nitrogen concentrations, therefore, an atmospheric blank should be determined.)
   a. Weigh ~0.15 g of ground 501-441 Sucrose into a 502-186 Tin Foil Cup and seal.
   b. Enter mass and identification information into Sample Login (F3).
   c. Transfer tin foil cup containing the sample to the appropriate position of the sample carousel.
   d. Initiate the analysis sequence (F5).
   e. Repeat steps 5a through 5d a minimum of three times.

   *Note: The nitrogen value obtained is considered the atmospheric blank and can be automatically compensated for using the CHN628 software. Refer to the operator's instruction manual for details regarding the setting of the atmospheric blank.*

## *TYPICAL RESULTS**

| | *Mass(g)* | *% Carbon* | *% Hydrogen* | *% Nitrogen* |
|---|---|---|---|---|
| Biomass | 0.1562 | 49.5 | 6.05 | 0.411 |
| Sample #1 | 0.1507 | 49.5 | 6.06 | 0.454 |
| Wood Pulp | 0.1573 | 49.5 | 6.00 | 0.416 |
| | 0.1528 | 49.5 | 6.05 | 0.447 |
| | 0.1534 | 49.5 | 6.05 | 0.451 |
| | ***Avg =*** | ***49.5*** | ***6.04*** | ***0.436*** |
| | ***s =*** | ***0.03*** | ***0.02*** | ***0.021*** |
| Biomass | 0.1495 | 50.0 | 6.05 | 0.110 |
| Sample #2 | 0.1498 | 49.9 | 6.06 | 0.100 |
| Wood Pulp | 0.1517 | 50.1 | 6.06 | 0.109 |
| | 0.1498 | 50.1 | 6.07 | 0.110 |
| | 0.1495 | 50.1 | 6.05 | 0.105 |
| | ***Avg =*** | ***50.0*** | ***6.06*** | ***0.107*** |
| | ***s =*** | ***0.08*** | ***0.01*** | ***0.004*** |
| Biomass | 0.1514 | 49.8 | 6.08 | 0.134 |
| Sample #3 | 0.1543 | 49.7 | 6.05 | 0.131 |
| Wood Pulp | 0.1517 | 49.8 | 6.09 | 0.132 |
| | 0.1519 | 49.8 | 6.07 | 0.130 |
| | 0.1506 | 49.8 | 6.08 | 0.130 |
| | ***Avg =*** | ***49.8*** | ***6.07*** | ***0.131*** |
| | ***s =*** | ***0.02*** | ***0.02*** | ***0.002*** |
| Atmospheric | 0.1547 | | | 0.018 |
| Blank | 0.1520 | | | 0.023 |
| | 0.1576 | | | 0.019 |
| | 0.1595 | | | 0.017 |
| | 0.1525 | | | 0.014 |
| | ***Avg =*** | | | ***0.018*** |
| | ***s =*** | | | ***0.003*** |

**Based on a single standard calibration with 0.15 g of 502-642 Phenylalanine weighed into 502-186 Tin Foil Cups. Results reported on a dry basis.*

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