

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 H₂O and CO₂, 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.



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.