

Carbon, Hydrogen, and Nitrogen in Coke

LECO Corporation; Saint Joseph, Michigan USA

Instrument: CHN628

Introduction

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

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. Helium 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.

Sampling and Sample Preparation

A representative, uniform sample is required. Samples should be prepared in accordance to ASTM D2013. Coal reference materials such as those offered by LECO and NIST are properly prepared.

Accessories

502-186 Tin Foil Cup or 502-040 Tin Capsule

Calibration

LECO 502-642 Phenylalanine, 502-092 EDTA, or other suitable pure compound

Analysis Parameters

Combustion Furnace Temperature	950°C
Afterburner Temperature	850°C

Element Parameters

	Nitrogen	Carbon	Hydrogen
Analyze	Yes	Yes	Yes
Baseline Delay Time	10	0	0
Minimum Analysis Time	40	20	40
Comparator Level	100.00	100.00	100.00
Endline Time	2	1	1
Conversion Factor	1.00	1.00	1.00
Significant Digits	5	5	5

IR Baseline Time	1
TC Baseline Time	10

Burn Profile

Burn Steps	Time	Furnace Flow
1	15	High
2	300	Medium
3	5	High

Macro Ballast Parameters

Ballast	
Equilibrate Time	30
Not Filled Timeout	600
Aliquot Loop	
Fill Time	40
Equilibrate Pressure Time	8
Fill Pressure Drop	250



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 blank using the last 5 results following the procedure outlined in the operator's instruction manual. Note: blank precision should be <0.001%.
3. Calibrate
 - a. Weigh ~0.1 g of pure compound calibration sample (EDTA, Phenylalanine, BBOT, etc.) into a 502-186 Tin Foil Cup and seal.
 - b. Enter sample mass and identification into Sample Login (F3).
 - c. Transfer sample to the appropriate position of the sample carousel.
 - d. Repeat steps 3a through 3c a minimum of five times.
 - e. Initiate the analysis sequence (F5).
 - 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 ~0.1 g of a pure compound different than the material used for calibration. For example, if Phenylalanine was used for calibration, verify the calibration using EDTA or BBOT.
4. Analyze Sample
 - a. Weigh ~0.08 to 0.1 g of coke sample into 502-186 Tin Foil Cup and seal.
 - b. Enter sample mass and identification into Sample Login (F3).
 - c. Transfer sample to the appropriate position on the sample carousel.
 - d. Initiate the analysis sequence (F5).

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.

Note: Coke should be analyzed "as received". Analytical values are corrected for moisture after analysis. Moisture should be determined within the same day the coke is analyzed.

Typical Results

(Based on single standard calibration with 0.1 g of 502-642 Phenylalanine)

Sample	Mass (g)	% Carbon	% Hydrogen	% Nitrogen
LECO 502-092	0.1002	41.07	5.60	9.55
EDTA Lot 1055	0.1021	41.01	5.55	9.55
41.06 ± 0.09% C	0.1020	41.02	5.54	9.55
5.55 ± 0.03% H	0.1016	40.95	5.54	9.54
9.56 ± 0.03% N	0.1009	40.97	5.55	9.55
	X =	41.00	5.56	9.55
	s =	0.05	0.03	0.004
LECO 502-684	0.0850	88.06	3.71	1.51
Petroleum Coke	0.0848	88.08	3.82	1.51
87.8 ± 1.7% C	0.0850	88.14	3.77	1.51
3.66 ± 0.42% H	0.0856	88.25	3.80	1.51
1.48 ± 0.24% N	0.0853	88.40	3.77	1.52
	X =	88.19	3.77	1.51
	s =	0.14	0.04	0.004
Metallurgical Coke	0.0728	87.34	0.22	1.04
LECO 502-683	0.0749	87.35	0.20	1.03
87.3 ± 2.0% C	0.0785	87.00	0.22	1.05
0.14 ± 0.10% H	0.0780	86.93	0.21	1.05
1.00 ± 0.22% N	0.0774	87.25	0.21	1.05
	X =	87.17	0.21	1.04
	s =	0.19	0.01	0.01

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